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ON HAZARDOUS MATERIALS

ANALYSIS METHODS

by

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A comprehensive annotated bibliography of analytical methods for 67 of the chemicals on the Environmental Protection Agency's Hazardous Substances List is presented. Literature references were selected and abstracts of analytical methods were compiled to facilitate rapid and accurate identification of the substances when discharged into the environment. Literature search procedures are outlined and general selection criteria for the analytical methods are discussed. Cross references are included for methods applicable to more than one of the hazardous substances.

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FOREWORD

The U.S. Environmental Protection Agency was created because of increasing public and government concern about the dangers of pollution to the health and welfare of the American people. Noxious air, foul water, and spoiled land are tragic testimonies to the deterioration of our natural environment. The complexity of that environment and the interplay of its components require a concentrated and integrated attack on the problem.

Research and development is that necessary first step in problem solution; it involves defining the problem, measuring its impact, and searching for solutions. The Municipal Environmental Research Laboratory develops new and improved technology and systems to prevent, treat, and manage wastewater and solid and hazardous waste pollutant discharges from municipal and community sources, to preserve and treat public drinking water supplies, and to minimize the adverse economic, social, health, and aesthetic effects of pollution. This publication is one of the products of that research and provides a most vital communications link between the researcher and the user community.

This report is a product of the above effort. It abstracts and cites published methods of chemical analysis for organic and inorganic hazardous materials. It thereby facilitates rapid selection of appropriate analytical methods pertaining to treatability studies, emergency response actions, and general pollution monitoring activities. This project is part of the continuing program of the Oil and Hazardous Materials Spills Branch, MERL-Ci, to assess and mitigate the environmental impact of oil and hazardous material pollution.

Francis T. Mayo, Director
Municipal Environmental Research
Laboratory

ABSTRACT

A comprehensive annotated bibliography of analytical methods for 67 of the chemicals on the Environmental Protection Agency's Hazardous Substances List is presented. Literature references were selected and abstracts of analytical methods were compiled to facilitate rapid and accurate identification of the substances when discharged into the environment. Literature search procedures are outlined and general selection criteria for the analytical methods are discussed. Cross references are included for methods applicable to more than one of the hazardous substances.

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BIBLIOGRAPHY ON HAZARDOUS MATERIALS ANALYSIS METHODS

I. Introduction

Section 311(b)(2)(A) of the Federal Water Pollution Control Act (Public Law 92-500) charges the Administrator of the Environmental Protection Agency (EPA) with the development of regulations designating as hazardous substances such elements and compounds that, when discharged into or upon navigable waters, present an imminent and substantial danger to the public health or welfare. Based on toxicological and discharge potential criteria, the EPA has designated 271 chemicals on its Hazardous Substances List.[1] Rapid and accurate identification of these chemicals is essential when an accident involving discharge of these substances into the environment occurs. To facilitate such identification, the Oil and Hazardous Materials Spills Branch of the EPA commissioned a review of the literature to determine the best methods of analysis for these chemical substances.

This report contains a comprehensive annotated bibliography and ancillary file of methods of analysis for 67 chemicals on the Hazardous Substances List. The compounds addressed herein fall into the following three major categories: 1) pesticides/herbicides, 2) organic compounds, and 3) inorganic compounds, excluding metal salts.

II. Development of the Bibliography

Selection of Target Chemicals

A target list of 100 compounds on the Hazardous Substances List was selected for tentative inclusion in the Bibliography on Hazardous Materials Analysis Methods. Emphasis was placed on organic compounds which retain their identity on admixture with cold water, as the analytical procedures were to be based on the assumption that samples are recovered from aqueous media. Several inorganic compounds, excluding metal salts, were then selected to yield the full complement of 100 compounds. Note that analytical methods for compounds appearing on both the "Priority Pollutants" list, also known as "Consent Decree" compounds, and the Hazardous Substances List have been addressed elsewhere and were therefore omitted from this bibliography.*

The Priority Pollutants list emanated from a court settlement[2], involving the EPA and several environmentally concerned plaintiffs, and commonly known as the "EPA Consent Decree". This judicial action required EPA to publish a list of toxic pollutants for which technology-based effluent limitations and guidelines would be required.

The EPA Project Officer divided the 100 chemicals into three different categories, based on priority. Hazardous substances thus determined to be of highest priority were addressed first. Within the budgeted manpower limitations for this bibliography, methods of analysis were assessed for the compounds in all but the lowest priority grouping. Table 1 comprises a list of the 67 compounds included in the final bibliography.

*EPA has proposed (Federal Register 44 (233), December 3, 1979) guidelines establishing test procedures for the analysis of pollutants. The proposed procedures include one or more analytical methods for each of the Priority Pollutants except bis(chloromethyl)ether, which decomposes rapidly in an aqueous medium.

Table 1. List of Compounds Included in Bibliography on Hazardous Material Analysis Methods

acetaldehyde
acetone cyanohydrin
allyl alcohol
amyl acetate
aniline
benzoic acid
benzonitrile
benzyl chloride
butyl acetate
butylamine
butyric acid
captan
carbaryl
carbon disulfide
chlorpyrifos
coumaphos
cresol
cyanogen chloride
2, 4-D acid
2, 4-D ester
diazinon
dicamba
dichlobenil
dichlone
2, 2-dichloropropionic acid
dichlorvos
diethylamine
dinitrobenzene
diquat
disulfoton
diuron
dodecylbenzenesulfonic acid
ethion
ethylenediamine
formic acid
fumaric acid
furfural
guthion
hydrofluoric acid
hydrogen cyanide
isoprene
kelthane
malathion
maleic acid
methoxychlor
methyl mercaptan

methyl methacrylate
methyl parathion
mevinphos
mexacarbate
naled
paraformaldehyde
parathion
phosphoric acid
propionic acid
pyrethrins
quinoline
resorcinol
strychnine
styrene
2, 4, 5-T acid
2, 4, 5-T ester
tetraethyl lead
trichlorfon
triethylamine
vinyl acetate
xylene

Literature Search Procedures

The primary data base for the literature searches was the Chemical Abstracts condensates files available through the Lockheed Dialog Retrieval Service. The Chemical Abstracts registry number for each compound was combined with key words such as analysis, identification, and determination to retrieve pertinent titles of technical publications. For most compounds, the search was conducted for publications which appeared during the years 1972 through 1978. The search was extended through 1970 (Volume 72 of Chemical Abstracts) if the recent literature did not yield a sufficient number of satisfactory references. In most cases for which a large number of appropriate references were available, the search was restricted to English-language publications. Through the Dialog system, access was gained not only to the chemical and biochemical journals but also to relevant sources such as NTIS and ASTM publications.

The titles thus retrieved were narrowed to the five to seven articles for each compound which appeared to be potentially most useful. Abstracts of these publications were read and considered. After the abstracts were examined, up to four papers which were expected to include appropriate analytical methods were ordered for each compound. Some of the papers thus selected did not contain the desired information. In those cases, additional papers were ordered, if available. To accommodate the report submission schedule, no papers were ordered after May 25, 1979. Up to three of the "best" methods, according to the criteria enumerated in the following section of this report, were included in the bibliography.

If no appropriate titles were retrieved, an effort was made to report a closely related compound, or a compound which can easily be derived from the desired compound. For example, formaldehyde was substituted for paraformaldehyde, the polymeric form of the former compound.

Several EPA laboratories were contacted by the Project Officer to determine whether work was underway in-house to develop analytical methods for the compounds under consideration and thus to avoid duplication of effort. The U. S. Coast Guard was similarly queried. This report does not appear to duplicate any existing reports or work in progress.

Selection Criteria for Analytical Methods

The formal selection criteria for the analytical methods were as follows:

- practicality of the method for rapid laboratory use (i.e., rapid and simple sample processing)
- occupational safety of prescribed reagents
- correctness of quantification data

- ability to identify the presence, and possibly the source, of the material

- practicality of the method in relation to the availability of common laboratory instruments (i.e., mass spectrometer; vapor phase and high-pressure liquid chromatographs; ultraviolet/visible, infrared, and nuclear magnetic resonance spectrophotometers; fluorescence and atomic absorption spectrometers, etc.)

Several other factors were considered in the selection of analytical methods. When three good analytical methods for aqueous samples were not available for a hazardous substance, the order of preference of sample source was 1) water, 2) sediment, 3) air, and 4) tissue. Superimposed on this ranking was a preference for English language articles to minimize translation problems. Variety in types of analytical methods was considered desirable. A rapid method for the mere detection of a compound might complement a more elaborate or time-consuming quantitative method. Whenever possible, methods were chosen employing different instrumentation techniques to circumvent delays caused by unavailability or temporary malfunctioning of a particular piece of equipment; consequently, two gas chromatographic methods and one spectrophotometric method would be preferred over three gas chromatographic methods. Lastly, if all other factors were deemed equal, the most recent publications were selected.

III. Description of the Bibliography

Organization and Information Content

The bibliography is organized alphabetically by compound name as indicated in the Hazardous Substances List. The entry for each compound includes the name, followed by alternate names, abstracts of up to three analytical methods, and, where applicable, cross references to methods for other compounds which identify or determine the compound of interest.

For each method, the general category of analytical method is reported (e.g., wet chemical, thin-layer chromatography, ultraviolet spectrophotometry, etc.), followed by the literature reference. The abstract itself is a summary not of the entire article but of the analytical procedure and includes the equipment and chemicals required for the analysis and general sample preparation measures. Information regarding maximum or minimum detection limits or sample size is given if these data were reported in the primary literature.

Although each analytical method is designated by the letter A, B, or C, the letters serve merely as labels for ease in cross-referencing and are not intended to connote the relative merits of the methods.

Utilization

The abstracts are designed to enable laboratory personnel to determine quickly which method is most appropriate for a given analysis problem. The lack of a suitable chromatographic column or reagent specified in the abstract would immediately be apparent, and cause a method to be eliminated from further consideration. Information regarding limits of detection and time required to perform the analysis is included, where available, to aid the analyst in selecting a method.

As many of the compounds included in the bibliography are closely related, the methods are frequently applicable to several compounds. Cross references are supplied in the following format: "see diazinon, method A; coumaphos, B; malathion, A."

An index of the compound names and all of the alternate names reported in the bibliographic headings provides a means of access to the analytical methods.

Limitations

When many references (more than 50) were located for a particular compound, only English-language publications were considered. Therefore, the "optimal" analytical methods might have been omitted if they appeared in foreign-language journals.

The selection was weighted toward recent papers in widely circulated journals, if all other considerations appeared to be equal. It is possible that a superior older method could have been excluded.

The analytical procedures were valid and current at the completion of literature survey. The user should be aware that the chemical literature changes and expands rapidly to reflect frequent innovations in sampling, wet-chemical procedures, and instrumentation.

IV. References

1. Federal Register 43(49), March 13, 1978

2. Natural Resources Defense Council v. Train, U. S. District Court, District of Columbia, Consent Decree, Natural Resources Defense Council, Inc., et al., v. Russell E. Train; Environmental Defense Fund et al. v. Russell E. Train; Citizens for a Better Environment et al. v. Russell E. Train; Natural Resources Defense Council, Inc. v. James A. Agee et al., Nos. 78-2153, 75-172, 75-1698, and 75-1267, June 8, 1976.

V. Bibliography

ACETALDEHYDE

- ethanal, ethyl aldehyde, acetic aldehyde

METHOD A - Wet Chemical and Visible Spectrophotometry

- CHARACTERISTIC DETECTION AND DETERMINATION OF ALIPHATIC ALDEHYDES
- Chem. Pharm. Bull. 1975, 23 (4), 891-894.
  S. Tagami, T. Nakamura, T. Nakano, and D. Shiho

- Acetaldehyde forms a red product on reaction with propionaldehyde (3-phenyl-2-quinoxalinyl)-hydrazone in 95% ethanol. Reaction is complete in 20 minutes and absorbance of the product is measured at 465 nm. The hydrazone is synthesized from propanal and 2-hydrazino-3-phenylquinoxaline. Effects of pH and temperature on the reaction between hydrazone and aliphatic aldehyde are discussed; aromatic aldehydes, glucose, and ketones do not react.
- Limits: 5×10^{-5} to 1×10^{-3} M acetaldehyde

METHOD B - Gas Chromatography

- RAPID SEPARATION OF LOWER ALIPHATIC CARBONYL COMPOUNDS BY GAS-LIQUID-SOLID CHROMATOGRAPHY
- J. Chromatogr. 1978, 152(2), 533-537
  Y. Hoshika and G. Muto

- Lower aliphatic carbonyl compounds are separated by gas chromatography, with flame ionization detection, on a column of 5% TCEP on 60/80 mesh Carbopack B. Relative retention times are reported for the compounds on several columns.
- Limits: < 300 ng

METHOD C - Titration

- MACRO- AND MICRO-DETERMINATIONS OF ACETALDEHYDE BY OXIDATION WITH PERMANGANATE IN ACID MEDIUM CONTAINING FLUORIDE IONS
- Indian J. Chem. 1977, 15A(6), 542-544
  K. A. Idriss, I. M. Issa, and M. M. Ghoneim

- A standard solution of permanganate in sulfuric acid/sodium fluoride quantitatively oxidizes acetaldehyde solution. The direct titration can be performed to a visual or potentiometric endpoint. The determination can also be made indirectly by back titration of excess permanganate against Tl(I) or Hg(I).
- Limits: 44 μg

Cross References - See furfural, method A; paraformaldehyde, C.

ACETONE CYANOHYDRIN

- 2-methyllactonitrile, alpha-hydroxyisobutyronitrile

METHOD A - Spectrofluorimetry

- DETECTION OF VARIOUS α - SUBSTITUTED NITRILES AND GEM HALONITROALKANES BY CHEMILUMINESCENCE
- Anal. Chim. Acta 1975, 77, 324-326
  H. W. Yurow and S. Sass

- Reaction of OOH^- with α-substituted nitriles gives an intermediate which oxidizes luminol to a chemiluminescent species. The nitrile is detected by mixing the sample in a spectrofluorimetric cell with alkaline luminol and 0.30% H_2O_2 in tetrasodium ethylenediaminetetraacetate and measuring intensity of light at 410 nm. Relative light intensities for 17 compounds are listed.
- Sample size: 0.2 ml at 1.0 mg/ml

METHODS B & C - Wet Chemical and Visible Spectrophotometry

- DETECTION OF ACETONE, α-HYDROXYISOBUTYRONITRILE, AND α-AMINOISOBUTYRONITRILE IN THE PRESENCE OF EACH OTHER: DETERMINATION OF α-HYDROXYISOBUTYRONITRILE
- Mikrochim. Acta 1974, (4), 759-764
  L. Légrádi

- Acetone cyanohydrin is detected in acetone by addition of p-nitroaniline diazonium salt solution and 2N sodium hydroxide to the acetone sample. The dark green color produced by acetone changes with concentration of the cyanohydrin. For quantitative determination, absorbance is measured at 440 nm after establishing a calibration curve.
- Limits: 10^{-4}% acetone cyanohydrin in acetone

- Copper sulfate solution and ammonia are added to an aqueous sample. Acetone cyanohydrin weakens or destroys the blue color of the copper tetraammine complex. For quantitative determination, absorbance is measured at 560 nm after establishing a calibration curve.
- Limits: 50 µg for detection.

ALLYL ALCOHOL

- o 2-propen-1-ol, 1-propenol-3, vinyl carbinol

METHOD A - Wet Chemical

- o ESTIMATION OF ALLYL ALCOHOL BY CHLORAMINE-T
- o Talanta 1973, 20(3), 349-351
  D. S. Mahadevappa and H. M. K. Naidu

- o Allyl alcohol in aqueous solution is estimated by mild oxidation with standard chloramine-T and iodometric back-titration of the excess oxidizing agent. The oxidation step is carried out in 0.5-1.0 M hydrochloric acid. The effects of pH and the presence of several foreign ions are discussed.
- o Sample size: 8-125 mg

AMYL ACETATE

- o amylacetic ester, iso-amylacetate, sec-amylacetate, t-amylacetate, pear oil, banana oil

METHOD A - Gas Chromatography

- o GAS CHROMATOGRAPHIC AND SENSORY ANALYSIS OF VOLATILES FROM CLING PEACHES
- o J. Agric. Food Chem. 1978, 26 (3), 725-732
  M. D. Spencer, R. M. Pangborn, and W. G. Jennings

- o Isoamyl acetate is one of 30 major volatile compounds identified in samples of cling peaches that are pureed, steam distilled, extracted, and analyzed by capillary gas chromatography. The column is coated with Carbowax 20M, and a flame ionization detector is used. Tridecane and octadecane serve as internal standards.
- o Limits: not given

METHOD B - Gas Chromatography and Mass Spectrometry

- o DETERMINATION OF BENZALDEHYDE IN WINES AND AMYL ACETATE AND ISOAMYL ACETATE IN LIQUEURS BY GAS-LIQUID CHROMATOGRAPHY AND CONFIRMATION BY MASS SPECTROSCOPY
- o J. Assoc. Off. Anal. Chem. 1975, 58(3), 582-584
  G. E. Martin, P. C. Buscemi, and B. B. Butts

- o Liqueur samples are diluted with water, extracted with pentane, dried, and analyzed by gas chromatography (with flame ionization detection) on a column of 10% SP-1000 on 100/120 mesh Chromosorb W (HP). Identification of amyl and isoamyl acetates is confirmed by mass spectrometry.
- o Limits: calibration of the gas chromatograph is linear for 0-50 ppm of amyl or isoamyl acetate

METHOD C - Gas Chromatography

- o GAS CHROMATOGRAPHIC RETENTIONS AS IDENTIFICATION CRITERIA
- o Anal. Biochem. 1977, 78(1), 244-251
  K. Yabumoto, W. G. Jennings, and M. Yamaguchi

- o Over 40 flavor essences recovered from muskmelon are identified through two-dimensional plots of Kováts indices measured on two wall-coated open tubular capillary gas chromatographic columns of different polarity. The coating materials are Carbowax 20M or methyl silicone SE-30, admixed with benzyltriphenylphosphonium chloride. Kováts indices are reported for butyl, isobutyl, amyl, and isoamyl acetates.
- o Limits: not given

ANILINE

- o aniline oil, phenylamine, aminobenzene, aminophen, kyanol

METHOD A - High-Pressure Liquid Chromatography

- o HIGH-PRESSURE LIQUID CHROMATOGRAPHY OF AROMATIC AMINES
- o J. Chromatogr. 1976, (119), 569-579
  P. R. Young and H. M. McNair

- o Fifty aromatic amines are separated by high-pressure liquid chromatography on various silica gels with detection at 254 nm. For aniline, a Corasil II column is used with chloroform as the mobil phase. A k' value of 0.40 is reported.
- o Sample size: 2-3 µg

METHOD B - Wet Chemical and Visible Spectrophotometry

- o DETECTION AND SPECTROPHOTOMETRIC DETERMINATION OF SOME AROMATIC NITROGEN COMPOUNDS WITH p-DIMETHYLAMINOCINNAMALDEHYDE
- o Anal. Chim. Acta 1976, 86, 309-311
  M. Qureshi and I. A. Khan

- o p-Dimethylaminocinnamaldehyde is used with Dowex 50W-X8 resin beads to detect aniline. A red color appears on the beads after heating for five minutes. Aniline is determined quantitatively with the aldehyde reagent, in a mixture of ethanol and hydrochloric acid. Color develops in 30 minutes, and absorbance is measured at 520 nm. A variety of compounds cause no interference in the procedure.
- o Limits: detection, 0.10 µg/ml; determination, 3-30 µg

METHOD C - Wet Chemical and Visible Spectrophotometry

- o A SIMPLE SIMULTANEOUS COLORIMETRIC DETERMINATION OF PRIMARY AND SECONDARY AMINES WITH FLUORESCAMINE
- o Anal. Lett. 1975, 8(7), 441-448
  V. Toome and K. Manhart

- o Primary and secondary amines react with fluorescamine to form, respectively, pyrrolinones or aminoenone chromophores with long wavelength absorption maxima. A solution of fluorescamine in dioxane is added to a solution of the amine or amine mixture in pH 8-8.5 phosphate buffer; reaction is complete within one minute, and spectrophotometric measurements can be made immediately. Tertiary amines, alcohols, acids, thiols, and aldehydes do not interfere.
- o Limits: For aniline, Beer's Law is obeyed for 0.00-0.05 µmol/ml.

Cross References - See benzonitrile, method A; ethylenediamine, C.

BENZOIC ACID

- benzenecarboxylic acid, phenylformic acid, dracylic acid

METHOD A - Wet Chemical and Ultraviolet Spectrophotometry

- DETERMINATION OF BENZOIC AND SORBIC ACIDS IN ORANGE JUICE
- Analyst (London) 1976, 101 (1198), 49-54
  T. Gutfinger, R. Ashkenazy, and A. Letan

- Benzoic and sorbic acids are determined under conditions which prevent their mutual interference. Aqueous or orange juice solutions of the two acid anions are acidified with orthophosphoric acid and steam distilled. A portion of the distillate is oxidized vigorously with potassium dichromate/sulfuric acid to destroy sorbic acid and volatile compounds. After redistillation, benzoic acid is determined spectrophotometrically at 225 nm against a standard curve.
- Limits: 40-400 ppm in water; 40-1000 ppm in orange juice

METHOD B - Wet Chemical

- MICRODETERMINATION OF BENZOIC AND SALICYLIC ACIDS WITH GUANIDINE CARBONATE AS A TITRANT
- Microchem. J. 1972, 61(2), 61-62
  A. K. Saxena

- Benzoic acid in a water-alcohol mixture is titrated to the bromcresol purple endpoint with standard guanidine carbonate solution.
- Limits: 0.061-0.610 mg with maximum error of $\pm$ 0.005 mg

Cross References - See formic acid, method A; phosphoric acid, A and B.

BENZONITRILE

- o phenyl cyanide, cyanobenzene

METHOD A - Anion-Exchange Chromatography

- o NON-AQUEOUS SOLVENT CHROMATOGRAPHY. II. SEPARATION OF BENZENE DERIVATIVES IN THE ANION-EXCHANGE AND n-BUTYL ALCOHOL SYSTEM
- o J. Chromatogr. 1972, 72, 187-191
  W. Funasaka, T. Hanai, K. Fujimura, and T. Ando

- o The distribution coefficients, K_d', of several mono- and disubstituted benzene compounds were determined by column chromatography (Amberlite CG-400 resin, type I, Cl-form) and ultraviolet spectrophotometry. K_d' values (in ml/ml) are: benzonitrile, 1.13; aniline, 4.14; o-xylene, 0.56; m-xylene, 0.55; p-xylene, 0.51; o-cresol, 4.26; m-cresol, 3.70; p-cresol, 3.39; o-dinitrobenzene, 3.62; m-dinitrobenzene, 2.06.
- o Limits: not given

METHOD B - Wet Chemical

- o MICROGRAM DETECTION OF NITRILES USING RESIN BEADS
- o Mikrochim. Acta 1977, 11-16
  S. Z. Qureshi and M. S. Rathi

- o Benzonitrile is detected via ion exchange resin beads (H^+ form) after hydrolysis with dilute sulfuric acid to produce ammonium ions. The ammonium ions are retained by the resin and detected by adding sodium hypochlorite and phenol solutions. N-unsubstituted amides react positively in the absence of sulfuric acid and can thus be distinguished from nitriles.
- o Limits: 0.002 M benzonitrile (10 µg in 0.05 ml)

Cross References - See dinitrobenzene, method B.

BENZYL CHLORIDE

METHOD A - Gas Chromatography

- THE GAS-LIQUID CHROMATOGRAPHY OF THE CHLOROETHANES AND CHLOROTOLUENES
- J. Chromatogr. 1973, 76(1), 101-113
  D. A. Solomons and J. S. Ratcliffe

- Mixtures of toluene, benzyl chloride, benzal chloride, and benzotrichloride are analyzed by gas chromatography, with a thermal conductivity detector, on a column of 8% dinonyl phthalate on 90/100 mesh Anochrom ADS. Benzal chloride is not completely resolved from benzotrichloride. A calibration curve is given for quantitative analysis.
- Sample size: 0.5 µl

METHOD B - Gas Chromatography

- SEPARATION OF BENZYL COMPOUNDS BY GAS-LIQUID CHROMATOGRAPHY
- J. Chromatogr. 1978, (157), 409-411
  A. E. Habboush and A. Z. Al-Rubaie

- A mixture of benzyl compounds is separated by gas chromatography (flame ionization detector). The following nine stationary phases are used, at 20% loading, on 80/100 mesh Chromosorb W as support: OV-1, OV-3, OV-11, OV-25, DEGS, DEGGLU, DEGA, EGTCP, and Carbowax 6000. The best separations are obtained with OV-11 and DEGGLU columns.
- Sample size: 0.25 g of each benzyl compound; 0.2 to 0.4 µl injection

METHOD C - Gas Chromatography

- QUANTITATIVE ANALYSIS OF MIXTURES OF TOLUENE AND ITS SIDE-CHAIN-CHLORINATED DERIVATIVES BY GAS CHROMATOGRAPHY
- J. Chromatogr. 1975, (114), 247-249
  R. Ramakrishnan and N. Subramanian

- Toluene and its side-chain-chlorinated derivatives are analyzed by gas chromatography with thermal conductivity detection on a column of 15% silicone oil DC 200 on Celite 545. Carbon tetrachloride is used as a standard. Relative retention times are reported.
- Limits: not given

BUTYL ACETATE

- acetic acid butyl ester

METHOD A - Gas Chromatography

- GAS-LIQUID CHROMATOGRAPHIC DETERMINATION OF SOLVENTS IN COMMERCIAL NAIL LACQUER PREPARATIONS
- J. Assoc. Off. Anal. Chem. 1977, 60 (3), 658-662
  M. J. Stutsman

- Dilution of commercial nail lacquer samples with isooctane effects precipitation of resins and pigments from the solvent mixture. After settling, n-butyl acetate is determined by temperature-programmed gas chromatography on a column of 10% PEG 20M on 70/80 mesh Gas-Chrom R. n-Propyl acetate is the internal standard, and a thermal conductivity detector is used.
- Limits: linear relationship of peak heights from 15-750 µg

METHOD B - Gas Chromatography

- A NEW PERSONAL SAMPLER FOR ORGANIC VAPORS
- J., Am. Ind. Hyg. Assoc. 1978, 39 (9), 701-708
  R. L. Bamberger, G. G. Esposito, B. W. Jacobs, G. E. Podolak, and J. F. Mazur

- Organic vapors are sampled by collection on an adsorption substrate contained within a small passive sampler worn by the worker. For n-butyl acetate determination, the sample is desorbed by carbon disulfide and analyzed by gas chromatography on a column of 10% FFAP. A flame ionization detector is used.
- Limits: 93% recovery at 37.5 ppm in air.

METHOD C - Gas Chromatography

- A CONVENIENT OPTIMIZED METHOD FOR THE ANALYSIS OF SELECTED SOLVENT VAPORS IN THE INDUSTRIAL ATMOSPHERE
- Amer. Ind. Hyg. Assoc., J. 1970, 31(2), 225-232
  L. D. White, D. G. Taylor, P. A. Mauer, and R. E. Kupel

- Solvent vapors from 10-l. air samples are adsorbed onto activated charcoal, desorbed by carbon disulfide, and analyzed by gas chromatography with flame ionization detection. A column of 10% FFAP on 80/100 mesh acid-washed DMCS Chromosorb W is used.
- Limits: For 0.5-2.0 TLV, recovery is greater than 90% for butyl acetate and for xylene.

Cross References - See amyl acetate, method C.

BUTYLAMINE

- 1-aminobutane

METHOD A - Ligand-Exchange Chromatography

- STUDIES ON LIGAND-EXCHANGE CHROMATOGRAPHY. V. GAS CHROMATOGRAPHIC SEPARATION OF LOWER ALIPHATIC AMINES BY LIGAND EXCHANGE
- J. Chromatogr. 1975, 114(1), 15-21
  K. Fujimura and T. Ando

- An inorganic cation exchanger, zirconium phosphate (ZP-1) in the Cu^{2+} form, is used as the stationary phase and nitrogen containing ammonia and water vapor as the mobile phase in the gas chromatographic separation of lower aliphatic amines. A flame ionization detector is used. The concentration of ammonia plays an important part in determining both resolution and retention times.
- Samples: aqueous solutions containing 5% of each amine.

METHOD B - Ion-Pair Chromatography

- ION-PAIR CHROMATOGRAPHY IN THE LOW CONCENTRATION RANGE BY USE OF HIGHLY ABSORBING COUNTER IONS
- J. Chromatogr. 1977, 142, 283-297
  J. Crommen, B. Fransson, and G. Schill

- A stationary phase containing a counter ion (naphthalene-2-sulfonate) of high absorbance is used in a high-performance system for ion-pair chromatography of non-UV-absorbing samples such as alkylamines. LiChrospher SI-100 is employed as the support phase. A good separation is obtained for isomeric six-carbon amines.
- Limits: approximately 1 ng

Cross References - See aniline, method C; diethylamine, A, B, and C; ethylenediamine, C.

BUTYRIC ACID

- butanoic acid, ethylacetic acid

METHOD A - Gas Chromatography

- GASH CHROMATOGRAPHIC ANALYSIS OF C_2-C_5 FATTY ACIDS IN AQUEOUS MEDIA USING CARBOPACK B-CARBOWAX 20M-PHOSPHORIC ACID
- J. Chromatogr. 1978, 150(1), 259-262
  J. C. DuPreez and P. M. Lategan

- A mixture of six C_2-C_5 fatty acids is separated by gas chromatography (flame ionization detection) on a column of 3% Carbowax 20M - 0.5% phosphoric acid on Carbopack B. Standard aqueous samples and a culture medium sample were analyzed.
- Limits: 8 ppm propionic or butyric acid.

METHOD B - Gas Chromatography

- DIRECT ANALYTICAL PROCEDURE FOR DETERMINATION OF VOLATILE ORGANIC ACIDS IN RAW MUNICIPAL WASTEWATER
- Water Res. 1978, 12(7), 437-446
  N. Narkis and S. Henfeld-Furie

- After addition of solid metaphosphoric acid to raw sewage, precipitated proteins and suspended solids are removed by centrifugation. The supernatant is injected onto a column of 20% Carbowax 20M and 3% phosphoric acid on 60-80 mesh acid-washed Chromosorb W. A flame ionization detector is used.
- Limits: approximately 5 mg carboxylic acid per liter.

Cross References - See formic acid, method B; propionic acid, A.

CAPTAN

- Orthocide-406, SR-406, Vancide-89

METHOD A - Liquid Chromatography

- CHLORINE-SELECTIVE DETECTION FOR LIQUID CHROMATOGRAPHY WITH A COULSON ELECTROLYTIC CONDUCTIVITY DETECTOR
- Anal. Chem. 1977, 49 (2), 326-331
  J. W. Dolan and J. N. Seiber
- High performance liquid chromatography on Vydac reversed phase packing is used with Coulson electrolytic conductivity detector for selective detection of poorly UV-absorbing organochlorine compounds in water and crop samples. Little or no interference is caused by compounds lacking chlorine or bromine heteroatoms.
- Limits: approximately 50 ng for lindane

METHOD B - Thin Layer Chromatography and Fluorometry

- SIMULTANEOUS DETERMINATION OF CAPTAN AND CAPTAFOL IN APPLES AND POTATOES BY THIN LAYER CHROMATOGRAPHY AND IN SITU FLUOROMETRY
- J. Assoc. Off. Anal. Chem. 1977, 60 (6), 1328-1330
  Y. Francoeur and V. Mallet
- A crop sample is homogenized and extracted, and captan and captafol are separated from each other and from co-extractives by thin layer chromatography on silica gel H impregnated with aluminum chloride. After the plates are sprayed with sodium chlorate and heated, fluorescence is measured directly.
- Limits: 0.02 ppm

METHOD C - Thin Layer Chromatography and Spectrofluorimetry

- THE FLUORIMETRIC DETECTION OF PESTICIDES ON ALUMINUM OXIDE LAYERS
- J. Chromatogr. 1976, 117 (1), 129-136
  G. E. Caissie and V. N. Mallet
- Fluorigenic properties of several pesticides on acidic and basic aluminum oxide thin layers are noted both before and after heat treatment of the chromatograms. Wavelengths and intensities of emissions are noted and compared with behavior of the compounds on silica gel plates.
- Limits: coumaphos, 0.02 µg; captan, 0.2 µg; guthion, 0.02 µg

Cross References - See dichlone, method A.

CARBARYL

- Sevin

METHOD A - Thin Layer Chromatography and Densitometry

- QUANTITATIVE DETERMINATION OF CARBARYL IN APPLES, LETTUCE, AND WATER BY DENSITOMETRY OF THIN LAYER CHROMATOGRAMS
- J. Assoc. Off. Anal. Chem. 1978, 61 (3), 616-620
  J. Sherma, A. J. Kovalchick, and R. Mack
- Carbaryl residues are extracted from water and separated on silica gel thin-layer plates. The plates are dipped successively in potassium hydroxide and p - nitrobenzene-diazonium fluoborate to develop color, scanned with a fiber optics densitometer, and compared with standards.
- Limits: 50 ng

METHOD B - Wet Chemical and Gas Chromatography

- GAS CHROMATOGRAPHIC DETERMINATION OF MICROAMOUNTS OF CARBARYL AND 1- NAPHTHOL IN NATURAL WATER AS SOURCES OF WATER SUPPLIES
- J. Chromatogr. 1977, 144 (1), 77-84
  K. Nagasawa, H. Uchiyama, A. Ogamo, and T. Shinozuka
- Water samples are extracted with methylene chloride, and carbaryl and its hydrolysis product, 1- naphthol, are separated from several known organochlorine contaminants on Amberlite XAD-8 resin. Heptafluorobutyryl derivatives are prepared and analyzed on three gas chromatographic columns with electron capture detection.
- Limits: 2.5-10 ppb for a 1- liter sample

METHOD C - Column Chromatography and High-Pressure Liquid Chromatography

- DIRECT ANALYSIS OF SOME CARBAMATE PESTICIDES IN FOODS BY HIGH-PRESSURE LIQUID CHROMATOGRAPHY
- J. Agric. Food Chem. 1977, 25(1), 211-212
  J. F. Lawrence
- Pesticide residues are extracted from crops and passed through a 2% deactivated Florisil column. The carbamate fraction is subjected to high-pressure liquid chromatography on LiChrosorb Si 60 with ultraviolet detection at 254 nm. Carbaryl and carbofuran require separation by reversed-phase chromatography with Permaphase ODS.
- Limits: carbaryl, 0.9 ng; zectran, 1.0 ng

Cross References - See diazinon, method B; mevinphos, B; mexacarbate, B and C; parathion, A; trichlorfon, B.

CARBON DISULFIDE

- carbon bisulfide, dithiocarbonic anhydride

METHOD A - Wet Chemical and Ultraviolet Spectrophotometry

- SPECTROPHOTOMETRIC DETERMINATION OF CARBON DISULPHIDE AS THE 1,2,3,4-THIATRIAZOL-5-THIOLATE ION
- Anal. Chim. Acta 1977, 92 (2), 393-397
  D. W. Franco and P. F. Romanelli

- A spectrophotometric method for the determination of carbon disulfide in water is based on the quantitative formation of the 1,2,3,4-thiatriazol-5-thiolate ion from carbon disulfide and azide ion. Absorbance is measured at 313 nm. Adjustments in procedure are described for analysis in the presence of cupric ion, other cations, sulfide, sulfite, nitrate, and nitrite.
- Limits: the system obeys Beer's Law for 2.3-8.6 µg carbon disulfide/ml.

METHOD B - Wet Chemical and Potentiometry or Atomic Absorption Spectrophotometry

- DETERMINATION OF CARBON DISULFIDE IN INDUSTRIAL ATMOSPHERES BY AN EXTRACTION-ATOMIC ABSORPTION METHOD
- Anal. Chem. 1975, 47(6), 942-944.
  B. M. Kneebone and H. Freiser

Aqueous solutions of carbon disulfide react with pyrrolidine to yield the dithiocarbamate, which subsequently reacts with copper (as cupric sulfate) to form a chelate. The disappearance of Cu^{2+} is monitored with a solid-state copper electrode. Alternatively, the chelate can be extracted into isoamyl acetate and determined by atomic absorption spectrophotometry.

- Limits: 7.0 µg CS_2 per sample

METHOD C - Wet Chemical and Gas Chromatography

- DETECTION OF CARBON DISULPHIDE (A DISULFIRAM METABOLITE) IN EXPIRED AIR BY GAS CHROMATOGRAPHY
- J. Chromatogr. 1974, 92(2), 442-444.
  J. Wells and E. Koves

Carbon disulfide is determined by gas chromatography after conversion to diethyldithiocarbamic acid. Expired air is bubbled into diethylamine in acetone and methyl chloride is added. After work-up, the residue is dissolved in n-butyl chloride and analyzed by gas chromatography (with flame ionization detection) on a column of 1.5% FFAP on 80/100 mesh Varoport 30.

- Limits: sub-microgram quantities can be determined

CHLORPYRIFOS

- Dursban

METHOD A - Capillary Gas Chromatography

- ANALYSIS OF ORGANOPHOSPHORUS PESTICIDES BY CAPILLARY GAS CHROMATOGRAPHY WITH FLAME PHOTOMETRIC DETECTION
- J. Chromatogr. 1976, 117 (1), 201-205
  W. Krijgsman and C. G. Van de Kamp

- Fifty-nine organophosphorus pesticides are analyzed by capillary gas chromatography on SE-30 with flame photometric detection. Relative retention times are given for two isothermal conditions and for temperature programming.
- Limits: approximately 100 pg

METHOD B - Thin Layer Chromatography and Fluorimetry

- METALLOFLUORESCENT INDICATORS AS SPRAY REAGENTS FOR THE IN SITU DETERMINATION OF ORGANOPHOSPHORUS PESTICIDES ON THIN-LAYER CHROMATOGRAMS
- Anal. Chim. Acta 1972, 60 (1), 13-23

- Water samples are extracted with organic solvent and chromatographed on silica gel N plates and sprayed with a quenched solution of palladium(II) - calcein or palladium-(II) - calcein blue. Organothiophosphorus insecticides are determined by displacement of palladium from its indicator complex, producing fluorescent spots on the plate.
- Limits: malathion, 10-50 ng; ethion, 10 ng; guthion, 10-20 ng; di-syston, 10 ng; chlorpyrifos, 50-100 ng

Cross References - See dichlone, method A; mexacarbate, A; trichlorfon, B.

COUMAPHOS

- Co-Ral

METHOD A - Thin Layer Chromatography

- DETECTION OF ORGANOPHOSPHORUS AND CARBAMATE PESTICIDES IN ADIPOSE TISSUE BY THIN-LAYER AND GAS-LIQUID CHROMATOGRAPHY
- N. Z. J. Sci. 1977, 20 (2), 221-224
  M. H. Clear, F. R. Fowler, S. R. B. Solly, and A. R. Ritchie

- Cholinesterase - inhibiting pesticides are extracted from adipose tissue, filtered at low temperature, and identified by thin-layer chromatography, treatment with bromine vapor, and visualization via cholinesterase spray solution. Confirmation is made by gas-liquid chromatography.
- Limits: 0.001-0.1 mg/kg adipose tissue

Cross References - See captan, method C; chlorpyrifos, A; dichlone, A.

CRESOL

- o cresylic acid, hydroxytoluene

METHOD A - Wet Chemical and Gas Chromatography

- o DETERMINATION OF TRACE PHENOLS IN WATER BY GAS CHROMATOGRAPHIC ANALYSIS OF HEPTAFLUOROBUTYRYL DERIVATIVES
- o J. Chromatogr. 1978, 156(1), 143-151
  L. L. Lamparski and T. J. Nestrick

- o Phenol and substituted phenols are derivatized with heptafluorobutyrylimidazole and determined by gas chromatography on a nitro-DEGS column. The derivatives are amenable to detection by electron capture. Sample preparation consists of benzene extraction of the aqueous medium and subsequent drying of the organic layer.
- o Limits: resorcinol and o-cresol are determined at concentrations $\geq$10 ng/ml.

METHOD B - Wet Chemical and Gas Chromatography

- o TRACE ANALYSIS OF PHENOLS IN WATER BY GAS CHROMATOGRAPHY
- o J. Fish. Res. Board Can. 1975, 32(2), 292-294
  D. A. J. Murray

- o With o-xylene as internal standard, aqueous samples of phenols, cresols, and xylenols are extracted with chloroform, concentrated, and reacted with Tri-Sil concentrate to form their trimethylsilyl derivatives. Gas chromatographic analysis is performed with flame ionization detection on a column of 5% Imol on Chromosorb W, acid washed. Calibration is linear in the 0-10 mg/l range.
- o Limits: cresols, 0.025 mg/l; xylenols, 0.050; phenol, 0.100.

METHOD C - Gas Chromatography

- o THE DIRECT-INJECTION GLC ANALYSIS OF XYLENOLS IN INDUSTRIAL WASTEWATERS
- o Bull. Environ. Contam. Toxicol. 1977, 17(6), 764-767.
  R. B. Baird, L. G. Carmona, and R. L. Jenkins

- o A direct aqueous injection method is developed for the gas chromatographic analysis of phenol, cresols,and xylenols in petroleum waste discharges. A column of 4% dinonylphthalate on 80/100 mesh Chromosorb G is used with flame ionization detection. o-Cresol is resolved from the mixture; m- and p-cresol appear as one peak.
- o Limits: samples in the mg/l range.

Cross References - See benzonitrile, method A; resorcinol, C.

CYANOGEN CHLORIDE

METHOD A - Wet Chemical and Visible Spectrophotometry

- o THE DETERMINATION OF CYANOGEN CHLORIDE
- o Analyst (London) 1972, 97(1158), 691-695
  P. L. Bailey and E. Bishop

- o Hydrochloric acid solutions of 1,4-diaminobenzene and pyridine react with cyanogen chloride to yield a solution which absorbs radiation at 515 nm. Interference from oxidizing chlorine species can be avoided by treatment of the contaminated cyanogen chloride solution with a mixture of hydrochloric acid, arsenic(III) oxide, and potassium bromide. Sulfur dioxide interference is removed by addition of barium nitrate to the colorimetric reagent.
- o Limits: cyanogen chloride solutions in the range 10^{-6} to 5×10^{-5} M

METHOD B - Wet Chemical and Gas Chromatography

- o SENSITIVE GAS CHROMATOGRAPHIC DETERMINATION OF CYANIDE
- o Anal. Chem. 1974, 46(7), 924-925
  J. C. Valentour, V. Aggarwal, and I. Sunshine

- o Cyanide is converted to cyanogen chloride by reaction with chloramine-T and analyzed by gas chromatography on a column of 7% Halcomid M-18 on Anakrom ABS. Response is linear up to 100 µg/ml, and the following ions cause no interference at 100 µg/ml: chloride, fluoride, bromide, iodide, and acetate.
- o Limits: 0.025 µg cyanide/ml

2,4-D ACID

- 2,4-dichlorophenoxyacetic acid

METHOD A - Wet Chemical and Gas Chromatography

- RAPID, SIMPLE PROCEDURES FOR THE SIMULTANEOUS GAS CHROMATOGRAPHIC ANALYSIS OF FOUR CHLOROPHENOXY HERBICIDES IN WATER AND SOIL SAMPLES
- J. Agric. Food Chem. 1978, 26 (3), 640-643
  B. A. Olson, T. C. Sneath, and N. C. Jain

- Water and soil samples containing 2,4-D, 2,4-DP, 2,4,5-T, and 2,4,5-TP are acidified, extracted into organic solvent, methylated with BF_3-methanol, and analyzed by gas chromatography with electron capture detector. Excellent separation of 5 compounds was obtained with the following columns: (1) 3% OV-17 on Gas-Chrom Q (80/100 mesh), (2) 3% Versamide - 900 on Supelcoport (80/100 mesh), (3) 15% QF-1/10% DC-200 on Chromosorb W (80/100 mesh).
- Limits: 0.001 ppm minimum quantitation level

METHOD B - Wet Chemical and Gas Chromatography

- GAS-LIQUID CHROMATOGRAPHIC METHOD WITH ELECTRON-CAPTURE DETECTION FOR THE DETERMINATION OF RESIDUES OF SOME PHENOXYACETIC ACID HERBICIDES IN WATER AS THEIR 2,2,2-TRICHLOROETHYL ESTERS
- J. Chromatogr. 1977, 136 (1), 105-111
  S. Mierzwa and S. Witek

- 2,4-D is determined by gas chromatography after sorption of an aqueous sample on Amberlite XAD-4, elution with benzene, and esterification with 20% 2,2,2-trichloroethanol in trifluoroacetic anhydride/sulfuric acid. Electron capture detection is used with a column of 10% DC-200 and 15% QF-1 on 80-100 mesh Gas-Chrom Q.
- Limits: 0.096 ppb 2,4-D in a 1- liter sample

METHOD C - Thin Layer Chromatography and Densitometry

- DETERMINATION OF CHLOROPHENOXY ACID HERBICIDES BY DENSITOMETRY ON THIN LAYER CHROMATOGRAMS
- Anal. Chim. Acta 1977, 91 (2), 259-266
  J. Sherma and J. Koropchack

- Chlorophenoxy acid herbicides and their salts are extracted from water with benzene, dried, and spotted on silica gel G plates that have been impregnated with a silver nitrate detection reagent solution. After ultraviolet irradiation, the spots are scanned by a fiber optics densitometer. Linear calibration curve is obtained in the 100-1000 ng range.
- Limits: 1 ppb

Cross References - See captan, method A; 2,4-D ester, B; dicamba, A, B, and C; dichlone, A; 2,2-dichloropropionic acid, B; 2,4,5-T acid, A; 2,4,5-T ester, A; trichlorfon, B.

2,4-D ESTER

- 2,4-dichlorophenoxyacetic ester

METHOD A - Wet Chemical and Gas Chromatography

- DETERMINATION OF INDIVIDUAL ESTERS OF 2,4-D and 2,4,5-T IN COMMERCIAL CROPS
- Pestic. Sci. 1977, 8 (2), 157-162
  H. E. Munro

- Individual esters of 2,4-D and 2,4,5-T are determined in purified crop samples by gas chromatography on a column of 9% XE60 or 2.5% NPGS on silanized Embacel. The samples are macerated, steam distilled into a hexane trap, extracted, and cleaned on a Florisil column. The following mixtures are separated: 2,4-D-butyl and 2,4,5-T-butyl esters; 2,4-D-ethyl, 2,4,5-T-butyl, and 2,4,5-T-isobutyl esters.
- Sample size: 1 µg each ester in 100 g plant material

METHOD B - Wet Chemical and High Pressure Liquid Chromatography

- ISOMER-SPECIFIC ASSAY OF ESTER AND SALT FORMULATIONS OF 2,4-DICHLOROPHENOXYACETIC ACID BY AUTOMATED HIGH PRESSURE LIQUID CHROMATOGRAPHY
- J. Assoc. Off. Anal. Chem. 1977, 60 (4), 868-872
  N. E. Skelly, T. S. Stevens, and D. A. Mapes

- 2,4-D is assayed in ester or salt formulations by in situ saponification (KOH/2-propanol/water), followed by separation by high pressure liquid chromatography on Partisil ODS, 10-25 µm. Results are reported as % 2,4-D acid equivalent. The method separates 2,4-D from known impurities and is applicable to 2,4-D esters of the following alcohols: methanol, 2-butoxyethanol, 1-butoxy-2-propanol, isooctanol, isobutyl alcohol, and n - butyl alcohol.
- Standard solution: 300 mg/25 ml

Cross References - See 2,4-D acid, method C; dichlone, A; dichlorvos, B; mevinphos, B; 2,4,5-T ester, A.

DIAZINON

- Dipofene, Diazitol, Basudin, Spectracide

METHOD A - Reaction Gas Chromatography

- REACTION GAS CHROMATOGRAPHIC ANALYSIS OF PESTICIDES. II. ON-COLUMN TRANSESTERIFICATION OF ORGANOPHOSPHATES BY METHANOL
- J. Agr. Food Chem. 1973, 21 (4), 621-625
  H. A. Moye

- Parathion and diazinon undergo on-column transesterification with methanol on a gas chromatographic column of 80/100 Porapak P or Q in which the first 6" is packed with sodium hydroxide-treated glass beads. Alkali flame ionization detection is used. Peaks were also obtained for transesterification with ethanol, 1-propanol, and 1-butanol. The organophosphates are separated from a representative carbamate.
- Limits: 1.0 ng

METHOD B - Gas Chromatography

- A MULTICLASS, MULTIRESIDUE ANALYTICAL METHOD FOR DETERMINING PESTICIDE RESIDUES IN AIR
- Arch. Environ. Contam. Toxicol. 1975, 3 (1), 55-71
  J. Sherma and T. M. Shafik

- Airborne pesticide residues are trapped by ethylene glycol, extracted by methylene chloride, and fractionated on silica gel. The fraction containing phosphate compounds is analyzed by gas chromatography with flame photometric detection on 5% OV-210 on Supelcoport, 80-100 mesh. Diazinon is well separated from malathion.
- Limits: 80 pg diazinon detected by gas chromatography; 40 pg, carbaryl; 460 pg, ethion; 400 pg, malathion; 320 pg, methyl parathion; 80 pg, mexacarbate; 40 pg, parathion

Cross References - See chlorpyrifos, method A; coumaphos, A; dichlone, A; dichlorvos, B; guthion, A and B; mevinphos, B; trichlorfon, B.

DICAMBA

- 2-methoxy-3,6-dichlorobenzoic acid

METHOD A - Wet Chemical and Gas Chromatography

- ANALYSIS OF PESTICIDES BY CHEMICAL DERIVATIZATION. III. GAS CHROMATOGRAPHIC CHARACTERISTICS AND CONDITIONS FOR THE FORMATION OF PENTAFLUOROBENZYL DERIVATIVES OF TEN HERBICIDAL ACIDS
- J. Assoc. Offic. Anal. Chem. 1976, 59 (3), 633-636
  A. S. Y. Chau and K. Terry

- Herbicidal acids are dissolved in benzene, converted to their pentafluorobenzyl ester derivatives by pentafluorobenzyl bromide and potassium carbonate, and analyzed by gas chromatography with electron capture detection. The best separation was obtained with a column of 3.6% OV-101 and 5.5% OV-210 on 80-100 mesh Chromosorb W.
- Limits: 0.8 µg

METHOD B - Wet Chemical and Thin-Layer Chromatography

- DETERMINATION OF SOME ACIDIC HERBICIDES BY THIN-LAYER CHROMATOGRAPHY
- J. Agric. Food Chem. 1971, 19 (6), 1181-1182
  A. Guardigli, W. Chow, and M. S. Lefar

- Six acid herbicides are extracted from tissue, cleaned by alkaline hydrolysis and extraction, converted to the nitro derivatives by sodium nitrate in concentrated orthophosphoric acid, and spotted on thin-layer chromatographic plates (precoated Merck silica gel F-254). Visualization is effected by spraying successively with a reducing agent (stannous chloride), a diazotizing agent, and the Bratton-Marshall reagent (N-(1-naphthyl)-ethylenediamine dihydrochloride).
- Limits: 0.5 µg (approximately 0.05 ppm in a crop residue)

METHOD C - Wet Chemical and Gas Chromatography

- ANALYSIS OF PESTICIDES BY CHEMICAL DERIVATIZATION. I. A NEW PROCEDURE FOR THE FORMATION OF 2-CHLOROETHYL ESTERS OF TEN HERBICIDAL ACIDS.
- J. Assoc. Offic. Anal. Chem. 1975, 58 (6), 1294-1301
  A. S. Y. Chau and K. Terry

- Ten herbicidal acids are purified by extraction and crystallization and subsequently converted to their 2-chloroethyl ester derivatives via dicyclohexyl carbodiimide and 2-chloroethanol. Gas chromatography with electron capture detection on one or more of the follwoing three columns allowed separation of the ten acids: 3.6% OV-101 and 5.5% OV-210 on 80-100 mesh Chromosorb W; 3% OV-225 on 80-100 mesh Chromosorb Q; 11% OV-17 and QF-1 on 80-100 mesh Chromosorb Q.
- Sample size required: 50-500 µg

Cross References - See 2,2-dichloropropionic acid, method B; 2,4,5-T acid, A.

DICHLOBENIL

- 2,6-dichlorobenzonitrile, 2,6-DBN

METHOD A - Gas Chromatography

- A MULTI-RESIDUE EXTRACTION PROCEDURE FOR THE GAS CHROMATOGRAPHIC DETERMINATION OF THE HERBICIDES DICHLOBENIL, DINITRAMINE, TRIALLATE AND TRIFLURALIN IN SOILS
- J. Chromatogr. 1974, 97 (1), 103-106
  A. E. Smith

- Four persistent herbicides are extracted from soil and analyzed by gas chromatography on 10% OV-1 on Chromosorb G-HP, 80-100 mesh. Electron capture detection is used.
- Limits: 0.05 ppm in soil

METHOD B - Wet Chemical and Gas Chromatography

- CONFIRMATION OF SOME ORGANONITROGEN HERBICIDES AND FUNGICIDES BY CHEMICAL DERIVATIZATION AND GAS CHROMATOGRAPHY
- J. Agric. Food Chem. 1976, 24 (6), 1236-1238
  J. F. Lawrence

- Several pesticides are extracted from corn and subjected to Florisil column cleanup. The fraction containing dichlobenil is methoxylated (methanolic sodium methoxide) and analyzed by gas chromatography on 4% SE-30/6% SP-2401 on Chromosorb W/HP, 80-100 mesh.
- Limits: 0.1 ppm

METHOD C - Bioassay

- BIOASSAYS FOR THE DETECTION OF HERBICIDES AND ALGICIDES IN WATER
- Meded. Fac. Landbouwwet. Rijksuniv. Gent 1977, 42 (2, Pt. 2), 1625-1634
  R. A. J. Bulcke and J. M. T. Strychers

- Dichlobenil inhibits root growth of garden cress seeds (Lepidium sativum L.). The seeds are placed on filter paper soaked with herbicide solution, and root length is measured after 48 hours. ED_{50} value is reported.
- Limits: qualitative detection in the range 0.02-0.14 ppm

Cross References - See 2,2-dichloropropionic acid, method B.

DICHLONE

- Phygon, dichloronaphthoquinone

METHOD A - Gas Chromatography

- RELATIVE RETENTION RATIOS OF NINETY-FIVE PESTICIDES AND METABOLITES ON NINE GAS-LIQUID CHROMATOGRAPHIC COLUMNS OVER A TEMPERATURE RANGE OF 170 TO 204°C IN TWO DETECTION MODES
- J. Assoc. Offic. Anal. Chem. 1975, 58 (5), 1037-1055
  J. F. Thompson, J. B. Mann, A. O. Apodaca, and E. J. Kantor

- Tables of relative retention data are given for 95 organochlorine and organophosphorus pesticides at temperatures from 170 to 204°C, in two-degree increments. Nine columns and two modes of detection (electron capture and flame photometric) were used. Two chromatograms are generally sufficient for tentative identification in multiresidue samples.

METHOD B - Column, Thin-Layer, and Gas Chromatography

- SEPARATION AND IDENTIFICATION OF 20 PESTICIDES IN THEIR MIXTURE
- Agr. Biol. Chem. 1973, 37 (8), 1959-1962
  K. Suzuki, K. Miyashita, H. Nagayoshi, and T. Kashiwa

- Dichlone, diuron, and 18 other pesticides undergo preliminary separation into 5 groups via column chromatography on silica gel. Each group is developed by two-dimensional thin-layer chromatography. Individual components are identified by ultraviolet spectroscopy and by gas chromatography on a column of 5% QF-1 on 80-100 mesh Gaschrom Q.
- Limits: approximately 1 mg

METHOD C - Thin-Layer Chromatography

- ELECTRON-DONOR-ACCEPTOR COMPLEXING REAGENTS IN THE ANALYSIS OF PESTICIDES. VI
- Mikrochim. Acta 1973, (5), 641-650
  J. D. MacNeil, R. W. Frei, and O. Hutzinger

- Dichlone is detected following thin-layer chromatography by spraying the chromatogram with a reagent that forms a π-complex with the pesticide. Colors are produced with both donors and acceptors. Mass spectrometry is used for confirmation.
- Sample size: 5 µg

2,2-DICHLOROPROPIONIC ACID

- Dalapon

METHOD A - Wet Chemical and Gas Chromatography

- DETERMINATION OF RESIDUES OF DALAPON IN SOIL BY GAS CHROMATOGRAPHY OF THE 1-BUTYL ESTER
- J. Chromatogr. 1975, 106 (2), 409-411
  E. G. Cotterill
- Dalapon is extracted from soil (using methanolic sodium hydroxide) and converted to its n-butyl ester via 1-butanol and sulfuric acid. The ester is analyzed with electron capture detection on a column of 2% OV-17 on Chromosorb WHP, 80-100 mesh.
- Limits: 0.05 ppm

METHOD B - Bioassay

- THE USE OF THREE SIMPLE, RAPID BIOASSAYS ON FORTY-TWO HERBICIDES
- Weed Res. 1971, 11(4), 257-262
  B. A. Kratky and G. F. Warren
- The following three bioassays were tested as primary screening methods on 42 herbicides: chlorella bioassay; root bioassay with sorghum, oat and cucumber; and shoot bioassay with sorghum and oat. A positive test is one in which inhibition is 50% or greater.
- Limits: dalapon, 30 ppm in soil; dicamba, dichlobenil, and 2,4-D, 1 ppm.

METHOD C - Gas Chromatography

- METHODS OF RESIDUE DETERMINATION AT THE BRITISH SUGAR CORPORATION RESEARCH LABORATORIES
- IIRB (Inst. Int. Rech. Betteravieres) 1973, 6(3), 138-153
  R. Parslow
- A sample of sugar beets is macerated with water, orthophosphoric acid, and phosphotungstic acid, extracted with diethyl ether, and analyzed by gas chromatography with electron capture detection. Column composition is 5% diethylene glycol adipate and 1% orthophosphoric acid on 60/85 mesh celite. Dalapon is confirmed by thin-layer chromatography on 60% Kieselghur G/40% Kieselgel G.
- Limits: 0.1 ppm by gas chromatography

DICHLORVOS

- o 2,2-dichlorovinyl dimethyl phosphate, Vapona

METHOD A - Thin Layer Chromatography

- o DETECTION AND DETERMINATION OF DICHLOROVOS (DDVP) IN TOXICOLOGICAL ANALYSIS
- o Fresenius' Z. Anal. Chem. 1977, 285 (1), 48
  S. N. Tewari and S. P. Harpalani

- o A biological sample is homogenized, extracted and spotted on a silica gel plate which is developed and sprayed with ethanolic thymol blue. The appearance of a violet color against a blue background (R_f 0.36) indicates dichlorvos.
- o Limits: 0.2 µg

METHOD B - Gel Permeation Chromatography and Gas Chromatography

- o AUTOMATED GEL PERMEATION CHROMATOGRAPHY CLEANUP OF ANIMAL AND PLANT EXTRACTS FOR PESTICIDE RESIDUE DETERMINATION
- o J. Assoc. Offic. Anal. Chem. 1976, 59 (1), 174-187
  L. D. Johnson, R. H. Waltz, J. P. Ussary, and F. E. Kaiser

- o Pesticide residues in fats and oils are separated by gel permeation chromatography on Bio-Beads SX-3. Toluene and ethyl acetate are used to elute sixteen nonionic chlorinated pesticides, three polychlorinated biphenyls, fourteen chlorophenoxy herbicide esters, and seven organophosphate insecticides. For gas chromatography on the pesticides, a column of 1.5% OV-17/2.0% OV-210 on Gas-Chrom Q (100-120 mesh) is used.
- o Sample Size: 0.5 g lipid

Cross References - See chlorpyrifos, method A; dichlone, A; trichlorfon, A and C.

DIETHYLAMINE

METHOD A - Wet Chemical and Gas Chromatography

- GAS CHROMATOGRAPHIC SEPARATION OF LOWER ALIPHATIC AMINES
- Anal. Chem. 1976, 48 (12), 1716-1717
  Y. Hoshika
- Thirteen aliphatic amines are analyzed by gas chromatography (with thermal conductivity detector) on a TENAX-GC column, 60/80 mesh. Primary amines are converted to Schiff bases by reaction with benzaldehyde; secondary and tertiary amines are analyzed in the form of their free amines.
- Sample size: approximately 10^{-7} mole

METHOD B - Wet Chemical, Gas Chromatography, and Mass Spectrometry

- THE SEPARATION AND IDENTIFICATION OF SOME ISOMERIC ALKYLAMINES AS THEIR TRIFLUOROACETYL DERIVATIVES
- J. Chromatogr. 1973, 82 (2), 373-376
  B. Crathorne and M. J. Saxby
- Trifluoroacetyl derivatives of 7 C_4 and 10 C_5 amines are prepared and analyzed by gas chromatography on a DEGS column with flame ionization detection. Diethylamine and t-butylamine have identical retention times but are distinguishable by mass spectrometry. n-Butylamine appears as a separate peak.

METHOD C - Wet Chemical, Thin-Layer Chromatography, and Fluorescence Spectroscopy

- FLUOROMETRIC DETERMINATION OF PRIMARY AND SECONDARY ALIPHATIC AMINES BY REACTION WITH 9-ISOTHIOCYANATOACRIDINE
- J. Pharm. Sci. 1973, 62 (8), 1370-1371
  A. DeLeenheer, J. E. Sinsheimer, and J. H. Burckhalter
- Primary and secondary aliphatic amines are determined by fluorescence spectroscopy of cyclic derivatives formed by reaction with 9-isothiocyanatoacridine. Wavelength for activation is 300 nm and for detection is 520 nm. Fluorescence is measured either directly on the reaction mixture or after thin-layer chromatographic separation on silica gel G.
- Limits: 0.2-0.5 nl/ml

Cross References - See aniline, method C.

DINITROBENZENE

- dinitrobenzol

METHOD A - Liquid Chromatography

- RETENTION BEHAVIOR OF DINITROBENZENE ISOMERS AND DIHYDROXYBENZENE ISOMERS ON OCTADECYL AND PHENYL BONDED PHASE PACKINGS
- J. Chromatogr. Sci. 1978, 16 (9), 438-442
  P. K. Tseng and L. B. Rogers

- Dinitrobenzene isomers are separated by liquid chromatography on Lichrosorb C_{18}, Bondapak C_{18}-Porasil B, or Bondapak phenyl/Porasil B with detection at 254 nm. Various methanol/water mixtures are used as eluent.
- Sample size: 30-80 µl

METHOD B - Gas Chromatography

- DETERMINATION OF TRACES OF ORGANIC COMPOUNDS IN THE ATMOSPHERE: ROLE OF DETECTORS IN GAS CHROMATOGRAPHY
- Anal. Chim. Acta 1978, 100, 45-52
  J. Mitchell, Jr. and H. D. Deveraux

- Gas chromatographic retention times and relative sensitivity data for various detectors are reported for 34 organic compounds. The column employed is 2% Carbowax 20M on 80/100 mesh GasChrom RA.
- Limits: nanogram range

METHOD C - Wet Chemical and Potentiometric Titration

- MICRODETERMINATION OF m-DINITRO COMPOUNDS WITH CYANIDE AND SILVER ELECTRODES
- Anal. Chem. 1977, 49(1), 45-48.
  S. S. M. Hassan

- m-Dinitro compounds react selectively and quantitatively with 0.2M potassium cyanide. Excess cyanide is titrated potentiometrically with 0.02M silver nitrate using a cyanide or silver/sulfide ion-selective electrode. No interference occurs from other classes of nitrogenous compounds.
- Sample size: 2-10 mg

Cross References - See benzonitrile, method A.

DIQUAT

- Aquacide, Dextrone, Reglone, Diquat dibromide

METHOD A - Wet Chemical and Gas Chromatography

- THE GAS CHROMATOGRAPHIC DETERMINATION OF PARAQUAT IN WATER
- Bull. Environ. Contam. Toxicol. 1972, 8 (6), 363-368
  C. J. Soderquist and D. G. Crosby

- Samples obtained from water are catalytically hydrogenated by sulfuric acid and PtO_2 in methanol, extracted into an organic solvent, and analyzed by flame-ionization gas chromatography on a column of 10% Triton X-100 plus 1% KOH on 70/80 AW, DMCS treated Chromosorb G. Diquat and paraquat can be analyzed simultaneously.
- Limits: <0.1 ppm

METHOD B - Pyrolysis and Gas Chromatography

- A RAPID METHOD FOR THE SIMULTANEOUS DETERMINATION OF PARAQUAT AND DIQUAT IN POND AND RIVER WATERS BY PYROLYSIS AND GAS CHROMATOGRAPHY
- Analyst 1975, 100 (1197), 848-853
  A. J. Cannard and W. J. Criddle

- Aqueous samples containing paraquat and diquat are pyrolyzed under carefully controlled conditions in a silica tube inserted into a Chemical Data Systems Pyroprobe 190. The pyrolysate is analyzed by gas chromatography on a column of 10% Carbowax 20M plus 2% KOH on Celite (80-100 mesh). Paraquat does not interfere with the diquat determination.
- Limits: 0.01 ppm

METHOD C - Wet Chemical and Gas Chromatography

- DETERMINATION OF DIQUAT AND PARAQUAT RESIDUES IN SOIL BY GAS CHROMATOGRAPHY
- J. Agr. Food Chem. 1974, 22 (5), 863-867
  S. U. Khan

- Paraquat and diquat residues are extracted from soil, hydrogenated catalytically over PtO_2, and analyzed by gas chromatography with flame ionization or alkali flame ionization detection. Of several columns that were explored, the best separation of the two compounds was obtained with 3% Carbowax 20M plus 1% KOH on 80-100 mesh Chromosorb WHP.
- Limits: 0.01 ppm

DISULFOTON

- o DI-syston

METHOD A - Wet Chemical, Gas Chromatography, Infrared Spectrophotometry

- o ANALYTICAL STUDIES OF DI-SYSTON FORMULATIONS
- o J. Assoc. Offic. Anal. Chem. 1972, 55 (5), 918-922
  J. C. Cavagnol

- o Several analytical methods for determining disulfoton are presented, including the following: (1) extraction, hydrolysis, and back titration; (2) infrared spectrophotometry at 610-750 cm^{-1}; (3) gas chromatography; (4) extraction and gravimetry; (5) colorimetry based on phosphorus.
- o Limits: none quoted

METHOD B - Wet Chemical and Visible Spectrophotometry

- o SEMIAUTOMATED ANALYSIS OF GRANULAR ORGANOPHOSPHORUS PESTICIDE FORMULATIONS
- o J. Agr. Food Chem. 1972, 20 (5), 959-963
  T. D. Talbott, J. C. Cavagnol, C. F. Smead, and R. T. Evans

- o Granular formulations of disulfoton are extracted with an acetic acid-bromine mixture and automatically analyzed colorimetrically for phosphorus via its molybdenum blue complex. Absorbance is measured at 829 nm.
- o Sample size: 1.65 g of 15% granules

Cross References - See captan, method A; chlorpyrifos, A and B; dichlone, A; dichlorvos, B; guthion, A and B; parathion, A.

DIURON

- DCMU, DMU

METHOD A - Wet Chemical and Gas Chromatography

- ANALYSIS OF SOME CARBAMATE AND UREA HERBICIDES IN FOODS BY GAS-LIQUID CHROMATOGRAPHY AFTER ALKYLATION
- J. Agric. Food Chem. 1975, 23 (6), 1106-1109
  J. F. Lawrence and G. W. Laver

- Carbamate and urea herbicides are extracted from crops with ethanol, partitioned between water and chloroform, alkylated with sodium hydride/methyl iodide, extracted with hexane, and analyzed by gas chromatography with a Coulson electrolytic conductivity detector in the nitrogen mode. Column composition is 4% SE-30/6% QF-1 on Chromosorb WHP (80-100 mesh.)
- Limits: approximately 0.005 ppm

METHOD B - Wet Chemical and Visible Spectrophotometry

- COLORIMETRIC DETERMINATION OF PHENYLAMIDE PESTICIDES IN NATURAL WATERS
- J. Assoc. Offic. Anal. Chem. 1972, 55 (6), 1276-1279
  M. A. El-Dib and O. A. Aly

- A simple rapid colorimetric procedure is given for phenylamide pesticides obtained from natural waters. The sample is hydrolyzed by sulfuric acid to produce corresponding anilines, which are extracted with chloroform, diazotized, and coupled with 1-naphthol to yield azo dyes. Absorbance at wavelengths of maximum absorption are measured on a visible spectrophotometer and compared with standards. Phenylamides may be analyzed in the presence of anilines.
- Limits: 0.02 mg/l.

METHOD C - Wet Chemical and Gas Chromatography

- A CHEMICAL CONFIRMATORY TEST FOR ORGANOPHOSPHORUS AND CARBAMATE INSECTICIDES AND TRIAZINE AND UREA HERBICIDES WITH REACTIVE NH MOIETIES
- J. Agric. Food Chem. 1975, 23 (2), 325-329
  R. Greenhalgh and J. Kovacicova

- Alkylation (sodium hydride/methyl iodide/dimethyl sulfoxide) followed by gas chromatography is used to confirm organophosphorus and carbamate insecticides and urea and triazine herbicides with NH or NH_2 moieties. An alkali flame ionization detector is used with a column of 100-120 Gas-Chrom Q coated with 4% SE-30/6% QF-1. The alkylated derivatives are thermally stable.
- Residues in crude plant and soil extracts can be confirmed at the sub part-per-million level.

Cross References - See dichlone, method B.

DODECYLBENZENESULFONIC ACID

- o laurylbenzenesulfonic acid

METHOD A - Potentiometry

- o ION SELECTIVE ELECTRODES RESPONSIVE TO ANIONIC DETERGENTS
- o Anal. Chem. 1974, 46(12), 1842-1844
  T. Fujinaga, S. Okazaki, and H. Freiser

- o Ion-selective electrodes responsive to anionic detergents are prepared by dipping a platinum wire into an appropriate coating mixture. For laurylbenzenesulfonate response, the coating consists of a 3:1 mixture of 10% polyvinyl chloride in cyclohexanone and a decanol solution of the quaternary ammonium salt formed by reaction of methyltricaprylammonium chloride and sodium laurylbenzenesulfonate. The electrode also responds to the lauryl sulfate and lauryl sulfonate ions.
- o Limits: calibration of the potential response curve is linear in the concentration range 10^{-3} - $10^{-5.5}$ M.

ETHION

- Nialate, ethyl methylene phosphorodithioate

METHOD A - Liquid-Liquid Extraction

- GUIDELINES FOR QUANTITATIVE LIQUID-LIQUID EXTRACTION OF ORGANOPHOSPHATE PESTICIDES FROM WATER
- ASTM Spec. Tech. Publ. 573 (Water Qual. Parameters, Symp., 1973), pp. 167-182 (1975)
  I. H. Suffet, C. Wu, and D. T. L. Wong

- An approach is developed for the quantitative analysis of aqueous samples of organophosphate pesticides, including ethion and parathion. Residue analysis includes liquid-liquid extraction, water and solvent phase separation, drying, and evaporation. The liquid-liquid extraction step is optimized. The p - values determined for pure water are applicable to natural waters.
- Recovery studies were conducted at the ng/l level.

Cross References - See chlorpyrifos, methods A and B; diazinon, B; dichlone, A; dichlorvos, B; guthion, A; mevinphos, B; trichlorfon, B.

ETHYLENEDIAMINE

- 1,2-diaminoethane

METHOD A - Wet Chemical and High-Performance Liquid Chromatography

- HIGH-PERFORMANCE LIQUID CHROMATOGRAPHIC SEPARATION AND QUANTITATION OF POLYFUNCTIONAL AMINES AS THEIR m-TOLUOYL DERIVATIVES
- J. Chromatogr. 1978, 154 (2), 219-225
  S. L. Wellons and M. A. Carey

- Polyfunctional aliphatic amines are determined at low levels in water, ammonia, and other amines by high-performance liquid chromatography of derivatives measurable with available ultraviolet detectors. Derivatization is accomplished with m-toluoyl chloride in pyridine. After extraction with methylene chloride, the amine derivatives are chromatographed on μ-Bondapak C_{18}.
- Sample size: 7 mg ethylene diamine before derivatization

METHOD B - Wet Chemical and High-Pressure Liquid Chromatography

- DETERMINATION OF DIAMINES AND POLYAMINES IN TISSUES BY HIGH-PRESSURE LIQUID CHROMATOGRAPHY
- J. Chromatogr. 1976, 124 (2), 277-285
  N. E. Newton, K. Ohno, and M. M. Abdel-Monem

- The dansyl (5-dimethylaminonaphthalene-1-sulfonyl) derivatives of five diamines obtained from tissue extracts are prepared and analyzed by high-pressure liquid chromatography on a column of Micropak CN-10. Fluorescence detection is used. Ethylene diamine is the internal standard.
- Sample size: 0.004 mmoles ethylene diamine

METHOD C - Wet Chemical and Visible Spectrophotometry

- SPECTROPHOTOMETRIC DETERMINATION OF MICROGRAM AMOUNTS OF AMINES WITH CHLORANIL
- Anal. Chim. Acta 1976, 85(1), 189-194
  T. S. Al-Ghabsha, S. A. Rahim, and A. Townshend

- Amines and chloranil react at 60-65°C in a pH 9 borate buffer to yield colored products. Wavelengths for absorbance measurements of the complexes are given.
- Limits: ethylene diamine, 2 ppm; n-butylamine, 2.5 ppm; aniline, 10 ppm.

FORMIC ACID

- methanoic acid

METHOD A - Wet Chemical and Gas Chromatography

- IMPROVED METHOD FOR ASSAY OF FORMIC ACID BY GAS-LIQUID CHROMATOGRAPHY
- J. Chromatogr, 1978, 151 (3), 374-378
  K. S. Bricknell and S. M. Finegold

- Formic acid obtained in a mixture of end-products produced by bacterial cultures is methylated with boron trifluoride/methanol and extracted with chloroform. The ester is converted to dimethylformamide by treatment with dimethylamine and analyzed by gas chromatography on a Resoflex column with thermal conductivity or flame ionization detector. Methyl esters of short-chain volatile fatty acids do not interfere, but free acetic acid does interfere with the analysis for dimethylformamide.
- Limits: 2 μmole/ml for thermal conductivity detector; 0.5 μmole/ml for flame ionization detector

METHOD B - Wet Chemical and Gas Chromatography

- DETERMINATION OF ORGANIC ACIDS OF LOW RELATIVE MOLECULAR MASS (C_1 to C_4) IN DILUTE AQUEOUS SOLUTION
- Analyst (London) 1974, 99(1175), 137-142
  P. O. Bethge and K. Lindström

- C_1-C_4 carboxylic acids are determined in low concentrations (5×10^{-5} M) in polluted water. Samples undergo ion exchange to remove metal cations and are then neutralized with tetra-n-butylammonium hydroxide, evaporated, dissolved in acetone, and converted to their benzyl esters by treatment with benzyl bromide. The esters are analyzed by gas-liquid chromatography, with flame ionization detection, on a column of 3% butane-1,4-diol succinate polyester on 120/140 mesh Chromosorb W, acid washed. n-Hexanoic acid is used as an internal standard.
- Limits: approximately 5×10^{-7} mole sample size

METHOD C - Wet Chemical and Gas Chromatography

- GAS CHROMATOGRAPHIC DETERMINATION OF SMALL AMOUNTS OF FORMIC ACID IN MIXTURES CONTAINING PHENOL, ACETONE, AND AROMATIC HYDROCARBONS
- J. Chromatogr. 1978, 148(1), 219-222
  W. Czerwiński and A. Stepień

- Formic acid is isolated from a mixture of aromatic hydrocarbons and acetone by extraction as its sodium salt. After acidification and esterification (methanol/anhydrous hydrogen chloride), the derivative is analyzed by gas chromatography on a column of 10% Carbowax 20M on 60-80 mesh Chromosorb W. A flame ionization detector is used. For formic acid concentrations between 0.005 and 0.1%, a linear calibration curve is obtained.
- Limits: 0.01-0.1% formic acid

FUMARIC ACID

- trans-butenedioic acid, trans-1,2-ethylenedi-carboxylic acid, boletic acid, allomaleic acid

METHOD A - Liquid Chromatography

- SEPARATION OF THE CITRIC ACID CYCLE ACIDS BY LIQUID CHROMATOGRAPHY
- Anal. Chem. 1978, 50 (11), 1420-1423
  V. J. Turkelson and M. Richards
- A variety of organic acids are analyzed by liquid chromatography on Aminex 50W-X4 cation exchange resin. The column eluent is monitored by ultraviolet spectrophotometry at 210 nm. As the column is operated in the hydrogen form, no regeneration of the resin is necessary.
- Limits: <1 ppm fumaric acid

METHOD B - Column and Thin-Layer Chromatography

- SEPARATION OF KREBS CYCLE ACIDS WITH BEADED DIETHYLAMINOETHYL CELLULOSE
- J. Assoc. Off. Anal. Chem. 1978, 61 (3), 751-752
  B. L. Bruinsma and D. Le Tourneau
- Organic acids are separated on a column of beaded diethylaminoethyl cellulose (Sephadex A-25, chloride form) without pH or solvent gradients. After solvent removal in vacuum, samples can be titrated for quantitative analysis. Identification of the acids is made by thin-layer chromatography on Brinkman cellulose MN300 and development by spraying with ribose-aniline reagent. The method has been used to determine acids produced in a culture medium.
- Sample size: 0.5 meq

METHOD C - Anion Exchange, Wet Chemical and Gas Chromatography

- ANALYSIS OF ORGANIC ACIDS IN FRUIT PRODUCTS BY ANION EXCHANGE ISOLATION AND GAS CHROMATOGRAPHIC DETERMINATION
- J. Assoc. Off. Anal. Chem. 1973, 56(5), 1257-1263
  D. W. Baker
- Alcoholic extracts of fruit products are passed successively through a cation exchange resin (Bio-Rad AG 50W-X2) and an anion exchange resin (Bio-Rad AG 1-X2). Acidic compounds trapped on the anionic column are eluted with formic acid/acetone, reacted with bis(trimethylsilyl) acetamide or bis-(trimethylsilyl)trifluoroacetamide to form trimethylsilyl ether-ester derivatives which are analyzed by gas chromatography with flame ionization detection. A column of 4% OV-1 on 100/120 mesh Gas-Chrom Q is used.
- Sample size: 1 mg

FURFURAL

- 2 - furaldehyde, pyromucic aldehyde

METHOD A - Wet Chemical and Gas Chromatography

- SENSITIVE GAS CHROMATOGRAPHIC DETERMINATION OF LOWER ALIPHATIC CARBONYL COMPOUNDS AS THEIR PENTAFLUOROPHENYL-HYDRAZONES
- J. Chromatogr. 1978, 152 (1), 224-227
  Y. Hoshika and G. Muto
- Twenty-seven lower aliphatic carbonyl compounds are derivatized with pentafluorophenylhydrazine and analyzed by gas chromatography. Columns and detectors employed are: 20% Ethofat 60/25 on Shimalite F (20-80 mesh), flame ionization; 5% SE-30 on Chromosorb W (60-80 mesh), flame ionization or electron capture; and glass capillary, PEG 20M, flame ionization.
- Sample size: 0.5 mmole carbonyl compound

METHOD B - Wet Chemical and Fluorescence Spectroscopy

- FLUORIMETRIC DETERMINATION OF AROMATIC ALDEHYDES WITH 2,2'-DITHIOBIS(1-AMINONAPHTHALENE)
- Anal. Chim. Acta 1978, 99 (2), 317-324
  Y. Ohkura, K. Ohtsubo, K. Zaitsu, and K. Kohashi
- A fluorimetric method for determining aromatic aldehydes is based on their reaction with 2,2'-dithiobis(1-aminonaphthalene) in the presence of tri-n-butylphosphine, sodium sulfite, and sodium phosphite. The method is selective for aromatic aldehydes; almost all other biologically important substances examined do not fluoresce at the concentrations employed. For furfural, excitation occurs at a wavelength of 380 nm and emission at 475 nm.
- Limits: 10 ng/ml

METHOD C- Wet Chemical and Ultraviolet Spectroscopy

- SPECTROPHOTOMETRIC DETERMINATION OF FURFURAL IN µg QUANTITIES USING ITS MOLYBDIC ACID COMPLEX
- Fresenius' Z. Anal. Chem. 1975, 276(4), 300
  V.K.S. Shukla and J. P. Sharma
- Furfural forms a colorless complex with molybdic acid. The complex absorbs at 331 nm and its solutions obey Beer's Law in the range of 100-700 µg.
- Limits: 100-700 µg

GUTHION

- o Gusathion, azinphos-methyl

METHOD A- Gas Chromatography

- o MULTIRESIDUE ANALYSIS OF FOURTEEN ORGANOPHOSPHORUS PESTICIDES IN NATURAL WATERS
- o J. Assoc. Off. Anal. Chem. 1974, 57 (5), 1033-1042
  B. D. Ripley, R. J. Wilkinson, and A. S. Y. Chau

- o Fourteen organophosphorus pesticides are determined in natural water samples by gas chromatography with flame photometric detection on a column of 11% OV-17/QF-1 on 80-100 mesh Chromosorb Q. Two extraction methods and two concentration methods are described.
- o Limits (sample size, 1ℓ): disulfoton, 0.005 ppb; guthion, 0.10 ppb; diazinon, methyl parathion, malathion, parathion, and ethion, 0.010 ppb.

METHOD B- Gas Chromatography

- o METHOD FOR ORGANOPHOSPHORUS PESTICIDES IN WATER AND WASTEWATER
- o Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater (Interim) 1978, 25-42
  U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio

- o An aqueous sample is extracted with methylene chloride in hexane, dried, concentrated, and analyzed by gas chromatography with flame photometric detector. Several organophosphorus pesticides are resolved on a column of 5% OV-210 or a mixture of 6% QF-1 and 4% SE-30 on 100/120 mesh Gas-Chrom Q. Cleanup and column variations are discussed.
- o Limits: 0.050-0.100 µg/ℓ for 1-ℓ. sample; 1 µg/ℓ for 100-ml. sample

Cross References - See captan, method C; chlorpyrifos, A and B; dichlone, A; mevinphos, B.

HYDROFLUORIC ACID

- o fluohydric acid

METHOD A - Potentiometry

- o NIOSH ANALYTICAL METHODS FOR SET L
- o NTIS PB-250159, 1976, 40-46
  Stanford Research Institute

- o Air samples are drawn through sodium hydroxide solution, diluted with additional sodium hydroxide solution, and further diluted with total ionic strength activity buffer. The diluted samples are analyzed using a fluoride ion specific electrode. Very large amounts of complexing metals such as aluminum may interfere.
- o Limits: method validated for 1.33-4.50 mg/m^3; probable useful range is 0.245-7.35 mg/m^3 for 45-liter sample

METHOD B - Coulometric titration

- o COULOMETRIC TITRATION OF HYDROFLUORIC ACID WITH A VITREOUS CARBON WORKING ELECTRODE
- o Anal. Chim. Acta 1975, 75(2), 478-485
  V. J. Jennings, A. Dodson, and D. Colbourne

- o A vitreous carbon cathode and silver anode are used in the coulometric determination of hydrofluoric acid. Nitrogen is bubbled through a potassium chloride solution, m-cresol purple solution is added as indicator, and current is passed through the solution until the color changes to determine the blank value. The hydrofluoric acid sample is added, and the electrolysis is continued until the indicator changes color again.
- o Limits: 10^{-4} - 10^{-5} moles of hydrogen fluoride

METHOD C - Potentiometry

- o AN AUTOMATIC POTENTIOMETRIC ANALYZER FOR ATMOSPHERIC HYDROGEN FLUORIDE DETERMINATIONS
- o Anal. Chim. Acta 1976, 85(2), 287-293
  M. Mascini

- o Hydrogen fluoride is collected from air by sodium carbonate and measured hourly by washing the absorber with a citrate buffer and determining fluoride with a fluoride-selective electrode. In the collector, a large filter impregnated with citric acid allows gaseous fluoride and gaseous fluoride adsorbed on particulate matter to pass, but retains particulate fluoride.
- o Limits: 0.1-15 $\mu g/m^3$ useful range

HYDROGEN CYANIDE

- hydrocyanic acid

METHOD A - Wet Chemical

- RAPID TEST PAPER METHODS FOR HYDROGEN CYANIDE IN THE ATMOSPHERE
- Ann. Occup. Hyg. 1971, 14(4), 289-294
  R. Hill, J. H. Holt, and B. Miller

- Test paper is treated with a mixture of p-nitrobenzaldehyde (in diacetone alcohol) and potassium carbonate. A reddish-purple color is obtained upon exposure of the paper to an atmosphere containing 10 ppm hydrogen cyanide. The reagent is stable and no interference is caused by the presence of a few hundred ppm of chlorine, ammonia, or nitrogen peroxide.
- Limits: approximately 5 ppm

METHOD B - Potentiometry

- DEVELOPMENT OF A SOLID SORPTION TUBE AND ANALYTICAL PROCEDURE FOR HYDROGEN CYANIDE IN THE WORKPLACE ATMOSPHERE
- NTIS PB-253 228, 1976, 1-20
  B. C. Cadoff and J. K. Taylor

- Atmospheric hydrogen cyanide is collected on flake sodium hydroxide in a sorption tube. After removal of the sample by water, the solution is analyzed potentiometrically with cyanide and saturated calomel electrodes. The method is rapid and relatively free of interferences.
- Limits: 10^{-5} - 10^{-3} M (concentrations greater than 10^{-3} M rapidly destroy the cyanide electrode)

METHOD C - Potentiometric Titration

- EVALUATION OF SAMPLING TECHNIQUES FOR CYANIDE EMISSIONS
- J. Am. Ind. Hyg. Assoc. 1978, 39(10), 832-835
  K. E. Williams, R. L. Bamberger, and G. G. Esposito

- Mixtures of air, carbon dioxide, and hydrogen cyanide are sampled through aqueous sodium hydroxide absorbents and the absorbents are analyzed potentiometrically (cyanide-specific electrode) by the method of standard additions. Hydrogen cyanide is determined in stack emissions containing up to 10% carbon dioxide. Absorbent temperature has no significant impact on results.
- Limits: samples containing 5 and 20 ppm hydrogen cyanide are analyzed successfully.

ISOPRENE

- 2-methyl-1,3-butadiene

METHOD A - Gas Chromatography

- GAS CHROMATOGRAPHIC ANALYSIS OF AMBIENT AIR FOR LIGHT HYDROCARBONS USING A CHEMICALLY BONDED STATIONARY PHASE
- Anal. Chem. 1974, 46(12), 1852-1854
  H. H. Westberg, R. A. Rasmussen, and M. Holdren

- Ambient air is sampled for light hydrocarbons (C_2-C_6) in an apparatus which is adaptable for mobile field units. The analysis is performed by gas chromatography on Durapak (n-octane/Porasil C). A sample trap immersed in liquid oxygen is preceded by a drying loop (anhydrous potassium carbonate).
- Limits: approximately 0.5 ppb.

METHOD B - Visible Spectrophotometry

- TETRACYANOETHYLENE π-COMPLEX CHEMISTRY. INDIRECT SPECTROPHOTOMETRIC DETERMINATION OF DIELS-ALDER-ACTIVE 1,3-DIENES
- Talanta 1973, 20(11), 1085-1096
  D. A. Williams and G. H. Schenk

- A colored π-complex between tetracyanoethylene (TCNE) and an aromatic compound such as naphthalene or mesitylene is destroyed by the Diels-Alder reaction of 1,3-dienes with TCNE. The decrease in absorbance of the complex in the presence of the 1,3-diene serves as the basis for the indirect spectrophotometric determination of isoprene.
- Limits: 2.5×10^{-5} M for the naphthalene-TCNE complex; 2.5×10^{-6} M for the mesitylene-TCNE complex.

METHOD C - Gas Chromatography

- AN IMPROVED COLUMN FOR BUTADIENE ANALYSIS
- J. Chromatogr. Sci. 1972, 10(12), 737-740
  J. W. Carson, J. D. Young, G. Legé, and F. Ewald

- Butadiene plant process streams and gaseous effluent from naphtha feedstock thermal cracking furnaces in ethylene plants are analyzed by gas chromatography. Two column sections are connected in series: 1) 20% dibutyl maleate on 60/80 Chromosorb P-NAW and 2) 10% bis(2-methoxyethoxy)-ethyl ether on the same support. C_4 and C_5 diolefins are separable.
- Limits: approximately 3% isoprene by weight.

KELTHANE

- di(p-chlorophenyl)trichloromethylcarbinol, DTMC, dicofol

METHOD A - Thin Layer Chromatography and Ultraviolet Spectrophotometry

- ISOLATION AND DETERMINATION OF CHLORINATED ORGANIC PESTICIDES BY THIN-LAYER CHROMATOGRAPHY AND THE APPLICATION TO TOXICOLOGICAL ANALYSIS
- J. Chromatogr. 1977. 131, 275-284
  S. N. Tewari and I. C. Sharma

- Twelve chlorinated organic pesticides are chromatographed on silica gel G plates, sprayed with a chromogenic reagent, and exposed to ultraviolet radiation. Results are given for 26 solvent systems and two chromogenic reagents. Densitometer response is linear from 1-10 µg.
- Limits: 0.05 µg Kelthane with ethanolic, ammoniacal silver nitrate spray; 0.5 µg Kelthane with 1% diphenylamine in ethanol spray

METHOD B - Thin Layer Chromatography and Ultraviolet Spectrophotometry

- THIN-LAYER CHROMATOGRAPHY OF DDT AND SOME RELATED COMPOUNDS ON ALUMINUM OXIDE CHROMATOPLATES
- J. Chromatogr. 1972, 64 (1), 135-145
  R. H. Bishara, G. S. Born, and J. E. Christian

- DDT and 11 related compounds are separated by two-dimensional thin-layer chromatography on aluminum oxide plates. The plates are activated under ultraviolet radiation, sprayed with a chromogenic reagent (ammoniacal silver nitrate plus 2-phenoxyethanol), and re-exposed to ultraviolet radiation.
- Limits: 0.05 µg Kelthane

Cross References - See captan, method A.

MALATHION

- Phosphothion

METHOD A - Gas Chromatography

- Ten NIOSH Analytical Methods, Set 1. U. S. NTIS, PB-271712, pp. 262-287 (1977)
  Stanford Research Institute
- Airborne malathion is collected on a filter, extracted with isooctane, and analyzed by gas chromatography (flame photometric detection) on a column of 1.5% OV-17 plus 1.95% OV-210 on 80/100 mesh GasChrom Q.
- Limits: 8-35 mg/m^3 in 106-ℓ sample

METHOD B - Thin Layer Chromatography and Visible Spectrophotometry

- DETERMINATION OF SOME ORGANOTHIOPHOSPHORUS PESTICIDES WITH PALLADIUM CHLORIDE, FOLLOWING THEIR SEPARATION BY THIN-LAYER CHROMATOGRAPHY
- Acta Pharm. Jugoslav. 1972, 22 (3), 91-96
  D. A. Jovanovic and Z. Prosic
- Malathion, methyl parathion, and bromophos are separated by thin-layer chromatography on Silica Gel F. After detection on a sprayed portion of the plate, the corresponding unsprayed portions are scraped off, dissolved in acetone and reacted with 0.05% palladium chloride solution. The solution is made up to volume and the optical density is determined at 320 nm.
- Limits: 0.25 ppm for food samples; 50-150 µg/100 ml water

Cross References - See chlorpyrifos, methods A and B; diazinon, B; dichlone, A; dichlorvos, B; guthion, A and B; mevinphos, A and B; parathion, A.

MALEIC ACID

- o cis-butenedioic acid, cis-1,2-ethylenedicarboxylic acid, toxilic acid

METHOD A - Liquid Chromatography

- o SEPARATION OF MONO- AND DICARBOXYLIC ACIDS BY LIQUID CHROMATOGRAPHY
- o J. Chromatogr. 1975, 115(1), 259-261
  M. Richards

- o A mixture of maleic, fumaric, citraconic, acrylic, and acetic acids is separated by liquid chromatography on a column of Aminex 50W-X4 (30-35 μm). The wavelength selected for monitoring is 210 nm.
- o Limits: 1μg/ml of each component, except acetic acid (100 μg/ml)

METHOD B - Potentiometry

- o ION-SELECTIVE ELECTRODE MEMBRANES RESPONSIVE TO MALEIC AND PHTHALIC ACIDS
- o Bull. Chem. Soc. Jap. 1973, 46(12), 3734-3737
  A. Jyo, M. Yonemitsu, and N. Ishibashi

- o Ion-selective electrodes responsive to hydrogen maleate anion are prepared via organic solvent solution of the anion with Crystal Violet or tris(bathophenanthroline)-iron(II) ion as the electrode membrane. Solvents successfully used for the membrane are nitrobenzene, 1,2-dichloroethane, and chloroform. Fumaric acid does not interfere in the maleic acid determination, and interferences from acetate, benzoate, and chloride are low.
- o Limits: electrode response is linear down to 10^{-4}-$10^{-4.5}$ M

METHOD C - Wet Chemical and Visible Spectrophotometry

- o SOLVENT EXTRACTION OF ANIONS WITH METAL CHELATE CATIONS - XVIII. SPECTROPHOTOMETRIC DETERMINATION OF MALEIC ACID IN THE PRESENCE OF FUMARIC ACID BY SOLVENT EXTRACTION WITH TRIS(1,10-PHENANTHROLINE)IRON(II) CHELATE CATIONS
- o Talanta 1972, 19(4), 415-422
  Y. Yamamoto, T. Kumamaru and M. Muranaka

- o Maleic acid is selectively extracted into nitrobenzene as the ion-association complex formed between the hydrogen maleate anion and the tris(1,10-phenanthroline)iron(II) cation. The complex is determined at 516 nm, and a linear relationship is obtained from 10^{-5} to 10^{-4} M maleic acid. The optimal pH range is 3-5.2. Maleic acid can be determined in the presence of up to 10^{-2} M fumaric acid.
- o Limits: 10^{-5}-10^{-4} M

METHOXYCHLOR

- DMDT, methoxy-DDT

METHOD A - Wet Chemical and Gas Chromatography

- EVALUATION OF XAD-2 FOR MULTIRESIDUE EXTRACTION OF ORGANOCHLORINE PESTICIDES AND POLYCHLORINATED BIPHENYLS FROM NATURAL WATERS
- J. Assoc. Off. Anal. Chem. 1977, 60 (1), 224-228
  J. A. Coburn, I. A. Valdmanis, and A. S. Y. Chau

- Organochlorine pesticides are extracted from water on XAD-2 macroreticular resin, cleaned on a column of deactivated alumina, and analyzed by gas chromatography (electron capture detector) on the following columns: (1) 4% SE-30 plus 6% OV-210 on 80-100 mesh Gas-Chrom Q; (2) 1.5% OV-17 plus 1.95% OV-210 on 80-100 mesh Gas-Chrom Q; (3) 3% OV-225 on 80-100 mesh Chromosorb W(HP).
- Limits: 0.1 µg/ml

METHOD B - Gas Chromatography

- RAPID PREPARATION OF MICRO SAMPLE AND GAS-LIQUID CHROMATOGRAPHIC DETERMINATION OF METHOXYCHLOR RESIDUES IN ANIMAL TISSUES AND WATER
- J. Assoc. Off. Anal. Chem. 1977, 60 (3), 690-695
  J. Solomon and W. L. Lockhart

- Water samples are extracted with n - hexane and analyzed by gas chromatography (electron capture detector) on a column of 2% SE-30 plus 3% QF-1 on 80-100 mesh Chromosorb W(HP). For tissue samples, extraction is followed by freezing out lipids and cleanup on a Florisil column.
- Limits: water samples, 1 ppb, 5-ml sample; animal tissue, 10 ppb, 0.1 g sample

METHOD C - Gas Chromatography

- METHOD FOR CHLORINATED HYDROCARBONS IN WATER AND WASTEWATER
- Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater (Interim) 1978, 7-24
  U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio

- An aqueous sample is extracted with methylene chloride in hexane, dried, concentrated, and analyzed by gas chromatography, with electron capture detection, on a column of 5% OV-210 on 100/120 mesh Gas-Chrom-Q. Cleanup and column variations are discussed.
- Limits: 0.050-0.100 µg/ℓ for 1-ℓ. sample; 1 µg/ℓ for 100-ml sample.

Cross References - See dichlone, method A; dichlorvos, B.

METHYL MERCAPTAN

- methanethiol, mercaptomethane, methyl sulfhydrate, thiomethyl alcohol

METHOD A - Gas Chromatography

- GAS CHROMATOGRAPHIC DETERMINATION OF SOME SULPHUR GASES AT THE VOLUMES PER MILLION LEVEL IN AIR USING TENAX-GC
- Analyst (London) 1978, 103(1225), 397-400
  D. S. Walker

- A method is reported for the gas chromatographic determination of hydrogen sulfide, carbonyl sulfide, sulfur dioxide, methyl mercaptan, and dimethyl sulfide either separately or in combination. A Tenax-GC column (35-60 mesh) and flame photometric detector are used.
- Limits: 1-100 ppm (volume)

METHOD B - Gas Chromatography

- SIMULTANEOUS GAS CHROMATOGRAPHIC SEPARATION OF VOLATILE ORGANIC SULPHUR COMPOUNDS AND C_1-C_4 HYDROCARBONS
- J. Chromatogr. 1974, 90(1), 218-222
  F. Raulin and G. Toupance

- Twenty-six volatile sulfur compounds and several C_1-C_4 hydrocarbons are analyzed simultaneously by gas chromatography on a column of Durapak OPN - Porasil C, 80-100 mesh. A flame ionization detector is used. The methanethiol peak is well resolved.
- Sample size: approximately 5 µg of each compound.

METHOD C - Gas Chromatography

- ANALYSIS OF SULFUR-CONTAINING GASES BY GAS-SOLID CHROMATOGRAPHY ON A SPECIALLY TREATED PORAPAK QS COLUMN PACKING
- Anal. Chem. 1975, 47(3), 543-545
  T. L. C. de Souza, D. C. Lane, and S. P. Bhatia

- A mixture of H_2S, COS, SO_2, MeSH, Me_2S, and Me_2S_2 is cleanly separated by gas chromatography on a column of acetone-washed Porapak QS. A flame photometric detector is used.
- Limits: approximately 5 ppm.

METHYL METHACRYLATE

- methacrylic acid methyl ester, methyl-2-methyl-2-propenoate

METHOD A - Proton Magnetic Resonance Spectroscopy

- DETERMINATION OF METHYL METHACRYLATE IN SURGICAL ACRYLIC CEMENT
- J. Pharm. Sci. 1976, 65(2), 280-283
  E. B. Sheinin, W. R. Benson, and W. L. Brannon

- Methyl methacrylate cement is quantitatively analyzed for its monomer content by proton magnetic resonance spectroscopy. The sample is dissolved in deuterated chloroform and integrals of the vinyl (δ 5.50-6.20) and methoxyl (δ 3.38-3.87) regions are determined in quintuplicate.
- Sample size: approximately 10 x 10 x 2mm

METHOD B - Gas Chromatography

- ANALYSIS OF THE INGREDIENTS AND DETERMINATION OF THE RESIDUAL COMPONENTS OF ACRYLIC BONE CEMENTS
- J. Biomed. Mater. Res. 1977, 11(4), 577-607
  G. M. Brauer, D. J. Termini, and G. Dickson

- Cured acrylic bone cement is examined for residual methyl methacrylate monomer and for water-leachable monomer by gas chromatography on a column of 25% Emulphor ON-870 on acid-washed, 80/100 mesh Chromosorb W. A flame ionization detector is used. For the water-leached sample, 0.02% aqueous n-hexyl alcohol is used as an internal calibration standard, and the height of the methyl methacrylate peak is compared to the alcohol peak. For the residual monomer determination, 0.1% ethyl methacrylate in methylene chloride is employed as internal standard.
- Limits: monomer content determined within 0.05% for 0.2-3% monomer range.

Cross References - See quinoline, method B.

METHYL PARATHION

- Nitrox-80

METHOD A - High Performance Liquid Chromatography

- DETERMINATION OF ETHYL AND METHYL PARATHION IN RUNOFF WATER WITH HIGH PERFORMANCE LIQUID CHROMATOGRAPHY
- Anal. Chem. 1977, 49 (11), 1551-1554
  D. C. Paschal, R. Bicknell, and D. Dresbach

- Aqueous samples are preconcentrated by treatment with XAD-2 resin, dissolved in acetonitrile, and chromatographed on a Partisil-ODS reverse phase column. Variable wavelength detection is used. Of 20 agricultural chemicals tested, only Fonofos interferes with the analysis for ethyl and methyl parathion.
- Limits: 2 ppb

METHOD B - Laser Excited Resonance Raman Spectroscopy

- DETECTION OF WATER POLLUTANTS BY LASER EXCITED RESONANCE RAMAN SPECTROSCOPY: PESTICIDES AND FUNGICIDES
- Appl. Spectrosc. 1978, 32 (1), 98-100
  R. J. Thibeau, L. Van Haverbeke, and C. W. Brown

- Raman spectra of aqueous samples of nitrobenzene-based pesticides and fungicides are recorded using the 488.0 and 457.9 nm Ar^+ laser lines.
- Limits: 7.0 ppm methyl parathion

Cross References - See chlorpyrifos, method A; diazinon, B; dichlone, A; dichlorvos, B; guthion, A and B; malathion, B; mevinphos, B; parathion, A; trichlorfon, B.

MEVINPHOS

- Phosdrin

METHOD A - Resin Trap and Gas Chromatography

- PORTABLE DEVICE WITH XAD-4 RESIN TRAP FOR SAMPLING AIRBORNE RESIDUES OF SOME ORGANOPHOSPHORUS PESTICIDES
- Anal. Chem. 1978, 50 (8), 1229-1231
  J. E. Woodrow and J. N. Seiber

- An XAD-4 resin trap is used for airborne samples of mevinphos, malathion, and parathion and compared with an ethylene glycol trap. After extraction from the resin, the sample is concentrated and analyzed by gas chromatography (alkali flame ionization detector) on a column of 3% OV-17 on 80/100 mesh Chromosorb G.
- Limits: 5 ng/m^3

METHOD B - Gas Chromatography

- EXTRACTION AND CLEANUP OF ORGANOCHLORINE, ORGANOPHOSPHATE, ORGANONITROGEN, AND HYDROCARBON PESTICIDES IN PRODUCE FOR DETERMINATION BY GAS-LIQUID CHROMATOGRAPHY
- J. Assoc. Off. Anal. Chem. 1975, 58 (5), 1020-1026
  M. A. Luke, J. E. Froberg, and H. T. Masumoto

- Thirty-one pesticides are extracted from produce and analyzed by gas chromatography with thermionic or electron capture detection; preliminary Florisil cleanup is necessary with electron capture detection. Several standard columns are employed.
- Limits (in ng): carbaryl, 106; parathion, 0.5; ethion, 0.76; guthion, 3.6; diazinon, 0.45

Cross References - See captan, method A; dichlone, A.

MEXACARBATE

- Zectran

METHOD A - High-Performance Liquid Chromatography

- HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY OF CARBAMATE PESTICIDES
- J. Chromatogr. Sci. 1976, 14 (12), 549-556
  C. M. Sparacino and J. W. Hines

- Thirty compounds are separated by high-performance liquid chromatography using both normal and reverse-phase modes. In the normal mode, the best separation for mexacarbate was obtained using a column of Si-10; for carbaryl, NH_2-10. Either μ-C_{18} or ETH was satisfactory in the reverse-phase mode.
- Limits: mexacarbate, 10.2 ng; carbaryl, 8.7 ng.

METHOD B - Wet Chemical and Gas Chromatography

- ESTERS OF SULFONIC ACIDS AS DERIVATIVES FOR THE GAS CHROMATOGRAPHIC ANALYSIS OF CARBAMATE PESTICIDES
- J. Agric. Food Chem. 1975, 23 (3), 415-418
  H. A. Moye

- Mexacarbate or carbaryl is reacted with 2,5-dichlorobenzene-sulfonyl chloride to yield the corresponding sulfonate esters which are analyzed by gas chromatography with tritium electron capture detection. Column packings are 5% LSX-3-0295 or UCW98 on 100-120 Hi-Performance Chromosorb W. Preliminary work has been carried out on flame photometric detection.
- Limits: 0.1 ppm for mexacarbate; 0.05 ppm for carbaryl

METHOD C - Thin-Layer Chromatography and Fluorescence Spectrophotometry

- ANALYSIS OF CARBAMATES AS FLUORESCENT 1-DIMETHYLAMINONAPHTHALENE-5-SULFONATE ESTERS
- Intern. J. Environ. Anals. Chem., 1972, 1, 317-325.
  J. F. Lawrence and R. W. Frei

- A fluorescent label is introduced into six N-methyl carbamates by reaction of their aqueous solutions with 1-dimethylamino-naphthalene-5-sulfonyl chloride (dansyl chloride). After extraction by n-hexane, the derivatives are subjected to thin-layer chromatography on silica gel G. Two fluorescent spots are obtained for each carbamate; emission maxima occur at approximately 530 nm.
- Limits: visual detection, 51 ng; instrumental detection, less than 1 ng per spot.

Cross References - See carbaryl, method C; diazinon, B.

NALED

- dibrom

Cross References - See chlorpyrifos, method A; dichlone, A.

PARAFORMALDEHYDE

- paraform, formagene, triformol, polymerized formaldehyde, polyoxymethylene

METHOD A - Wet Chemical and Gas Chromatography

- GAS CHROMATOGRAPHIC DETERMINATION OF METHANAL TRACES IN PRESENCE OF OTHER VOLATILE CARBONYL COMPOUNDS
- J. Chromatogr. 1978, (153), 530-531
  E. Merat

- Methanal is determined in concentrated liquid apple and smoke flavors in the presence of other volatile carbonyl compounds by derivatization with 2,4-dinitrophenylhydrazine. The 2,4-dinitrophenylhydrazone is extracted into benzene and analyzed by gas chromatography, with flame ionization detection, on a column of 4% SE-30 plus 4% OV-17 on 100/120 mesh Chromosorb W HP. The same support is also used with 10% OV-101 stationary phase.
- Limits: 10 ppm

METHOD B - Gas Chromatography

- DETERMINATION OF THE CYCLIC TRIMER OF FORMALDEHYDE BY GAS-LIQUID CHROMATOGRAPHY
- Analyst (London) 1978, 103(1230), 979-982
  V. B. Kapoor, S. K. Chopra, and S. C. Vishnoi

- A mixture of formaldehyde, 1,3,5-trioxan, methanol, water, and benzene is separated by gas chromatography, with thermal conductivity detection, on a column of 20% polyoxyethylene sorbitan monooleate on 60/80 mesh Chromosorb P.
- Sample size: 1.0 µℓ

METHOD C - Wet Chemical and Gas Chromatography

- GAS CHROMATOGRAPHIC SEPARATION OF CARBONYL COMPOUNDS AS THEIR 2,4-DINITROPHENYLHYDRAZONES USING GLASS CAPILLARY COLUMNS
- J. Chromatogr. 1976, 120(2), 379-389
  Y. Hoshika and Y. Takata

- 2,4-Dinitrophenylhydrazone derivatives of a mixture of 10 aliphatic aldehydes are separated by capillary gas chromatography, using SF-96 stationary phase and flame ionization detection. A 30-ℓ. car exhaust sample is condensed and dissolved in ethanol. The 2,4-dinitrophenylhydrazone derivatives are crystallized overnight, purified, and dissolved in acetone. Anthracene is employed as internal standard.
- Limits: exhaust sample containing 2.3 ppm formaldehyde and 1.5 ppm acetaldehyde is well resolved.

Cross References - See furfural, method A.

PARATHION

- DNTP, Niran

METHOD A - Gas Chromatography

- GAS-LIQUID CHROMATOGRAPHY OF AQUEOUS-ALCOHOL SOLUTIONS FOR INSECTICIDE RESIDUE ANALYSIS
- J. Assoc. Off. Anal. Chem. 1978, 61 (4), 837-840
  W. W. Sans

- Aqueous solutions of 20 insecticides are centrifuged, diluted with methanol and analyzed by gas chromatography (alkali flame detection) on a column of 10% QF-1 on 100-120 mesh Aeropak 30.
- Limits (in ng): parathion, 0.03; methyl parathion, 0.03; malathion, 0.03; carbaryl, 3.1; disulfoton, 0.01

METHOD B - Gas Chromatography

- ANALYTICAL METHOD FOR PARATHION IN AIR
- Health Lab. Sci. 1976, 13(1), 73-77
  D. F. Adams, J. O. Frohliger, D. Falgout, A. M. Hartley, J. B. Pate, A. L. Plumley, F. P. Scaringelli, and P. Urone

- Parathion is removed from an air sample by passing a measured volume through an impinger containing ethylene glycol. After extraction into hexane, the sample is analyzed by gas chromatography on a column of 4% SE-30/6% OV-210 on 80/100 mesh Chromosorb W. A flame photometric detector is used.
- Limits: 5-250 $\mu g/m^3$

Cross References - See chlorpyrifos, method A; diazinon, A and B; ethion, A; guthion, A; mevinphos, A and B; trichlorfon, B.

PHOSPHORIC ACID

- orthophosphoric acid

METHOD A - Catalytic Thermometric Titrimetry

- THE DETERMINATION OF WEAK ACIDS IN AQUEOUS SOLUTION BY CATALYTIC THERMOMETRIC TITRIMETRY
- Talanta 1976, 23(1), 73-76
  E. J. Greenhow and A. A. Shafi

- Weak acids are determined in aqueous solution with dimethyl sulfoxide as co-solvent and acrylonitrile as indicator. The titrant is potassium hydroxide in propan-2-ol. A sharp rise in temperature occurs at the endpoint due to heat evolved in the anionic polymerization of the indicator.
- Limits: for a 0.1 mmole sample, the concentration limits are: phosphoric acid, 1%; benzoic acid, 2%; resorcinol, 10%.

METHOD B - Gas Chromatography and Mass Spectrometry

- ORGANIC ACID PROFILES OF HUMAN TISSUE BIOPSIES BY CAPILLARY GAS CHROMATOGRAPHY-MASS SPECTROMETRY
- J. Chromatogr. 1977, 142, 497-503
  S. I. Goodman, P. Helland, O. Stokke, A. Flatmark, and E. Jellum

- Tissue specimens are homogenized and extracted to yield a mixture of acids, which are converted to their trimethylsilyl derivatives. The derivatives are analyzed by combined gas chromatography (glass capillary column coated with SE-30) and mass spectrometry.
- Limits: 5mg tissue sample

METHOD C - Wet Chemical

- MICRODETERMINATION OF THE PHOSPHATE ION USING A NEW REACTION
- Mikrochim. Acta 1974, (4), 561-566.
  S. S. M. Hassan and S. A. I. Thoria

- Phosphoric acid and primary and secondary phosphates are determined by indirect iodometric titration. Phosphate displaces iodate from calcium iodate in the presence of ammonia to form the less soluble calcium ammonium phosphate. The soluble iodate is determined by addition of potassium iodide and titration of the liberated iodine with sodium thiosulfate. Fluoride, silicate, sulfate, and arsenate interfere with the analysis; chloride, bromide, iodide, and nitrate do not interfere.
- Sample size: 2-10 mg

PROPIONIC ACID

- propanoic acid, methylacetic acid, ethylformic acid

METHOD A - Wet Chemical and Gas Chromatography

- GAS-LIQUID CHROMATOGRAPHY OF SHORT-CHAIN FATTY ACIDS ON DEXSIL 300GC
- J. Chromatogr. 1972, 74(2), 335-338
  M. A. Lambert and C. W. Moss
- A mixture of twelve C_1-C_7 fatty acids is derivatized via reaction with trifluoroacetic anhydride in boron trifluoride/butanol. The esters are analyzed by gas chromatography (flame ionization detection) on a column of 15% Dexsil 300GC on 80-100 mesh DMCS-treated Chromosorb W.
- Limits: 2.5 µg

Cross References - See butyric acid, methods A and B; formic acid, B.

PYRETHRINS

- Pyrethrin I, Pyrethrin II

METHOD A - Gas Chromatography and Mass Spectrometry

- SEPARATION AND ANALYSIS OF THE PYRETHRINS BY COMBINED GAS-LIQUID CHROMATOGRAPHY-CHEMICAL IONIZATION MASS SPECTROMETRY
- J. Assoc. Off. Anal. Chem. 1977, 60 (3), 685-689
  R. L. Holmstead and D. M. Soderlund
- The six naturally occurring pyrethrin esters and pyrethrins I and II are separated by gas chromatography on a column of 5% OV-25 on 60-80 mesh Chromosorb W. Chemical ionization mass spectra are also reported.
- Limits: 114 ng total to detect all compounds; 11.4 ng total to detect Pyrethrin I

METHOD B - Gas Chromatography and Mass Spectrometry

- ANALYTICAL STUDIES OF PYRETHRIN FORMULATIONS BY GAS CHROMATOGRAPHY III. ANALYTICAL RESULTS ON INSECTICIDALLY ACTIVE COMPONENTS OF PYRETHRINS FROM VARIOUS WORLD SOURCES
- J. Chromatogr. 1974, 90 (1), 119-128
  Y. Kawano, K. H. Yanagihara, and A. Bevenue
- Matrix-programmed flame ionization gas chromatography is used to separate the components of pyrethrum extracts on a column of 2.5% XE-60 on 60-100 mesh Chromosorb W after Florisil column cleanup of the crude extracts. Mass spectra in the m/e 50-190 range are given.
- Sample size: 80-120 µg

METHOD C - Gas Chromatography

- USE OF PROGRAMMED TEMPERATURE IN ANALYTICAL DETERMINATION OF PYRETHRINS BY ELECTRON CAPTURE DETECTOR
- Pyrethrum Post 1971, 11 (1), 29-31
  P. Tetenyi, E. Hethelyi, T. Okuda, and I. Szilagyi
- Both isothermal and programmed temperature gas chromatographic methods are used to analyze pyrethrum extracts. An electron capture detector is employed with a column of 3% NPGS on silanized Chromosorb W, 60/80 mesh.
- Sample size: 5 µg pyrethrum extract in 3 µl

QUINOLINE

- 1-benzazine, benzo(b)pyridine, leuocoline, chinoleine, leucol

METHOD A - High-Pressure Liquid Chromatography

- SEPARATION OF AZA-ARENES BY HIGH-PRESSURE LIQUID CHROMATOGRAPHY
- J. Chromatogr. Sci. 1977, 15 (1), 32-35
- M. Dong and D. C. Locke

- Aza-arenes are separated in 20 minutes by high-pressure liquid chromatography using reversed-phase or adsorbent packings. Identification is made by UV (254 nm) or fluorescence spectrophotometry. The method is applied to the analysis of airborne particulate matter.
- Limits: 1 ng

METHOD B - Column and Gas Chromatography

- USE OF MACRORETICULAR RESINS IN THE ANALYSIS OF WATER FOR TRACE ORGANIC CONTAMINANTS
- J. Chromatogr. 1974, 99, 745-762
- G. A. Junk, J. J. Richard, M. D. Grieser, D. Witiak, J. L. Witiak, M. D. Arguello, R. Vick, H. J. Svec, J. S. Fritz, and G. V. Calder

- Trace organics are isolated from water samples by sorption on XAD-2 or XAD-4 macroreticular resins and elution with diethyl ether. After concentration, the eluent is analyzed by gas chromatography (flame ionization detector) on a column of 5% OV-1 on 80-100 mesh DMCS-treated Chromosorb W.
- Limits: recovery of quinoline at the 10-100 ppb level is 84%; methyl methacrylate, 35%.

METHOD C - High-Performance Liquid Chromatography

- HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY WITH Ag^+ COMPLEXATION IN THE MOBILE PHASE
- J. Chromatogr. 1978, 149, 417-430
- B. Vonach and G. Schomburg

- Six N-heterocycles, including quinoline, and standard nitrogen-free polyaromatics are separated by high-performance liquid chromatography on Nucleosil 5 C 18 with a polar mobile phase containing silver perchlorate.

Cross References - See strychnine, method C.

RESORCINOL

- o resorcin, 1,3-benzenediol, meta-di-hydroxybenzene

METHOD A - Wet Chemical and Gas Chromatography

- o N-METHYLIMIDAZOLE-CATALYZED ACETYLATION OF HYDROXY COMPOUNDS PRIOR TO GAS CHROMATOGRAPHIC SEPARATION AND DETERMINATION
- o Anal. Chem. 1979, 51(1), 27-30
  R. Wachowiak and K. A. Connors

- o Alcohols, phenols, glycols, and sugars are acetylated with acetic anhydride in the presence of N-methylimidazole and determined by gas chromatography with flame ionization detection. For phenols, the stationary phase is 5% Carbowax 20M or 5% OV-17 on Chromosorb W - AW, DMCS, and ethylene glycol diacetate is employed as an internal standard.
- o Sample size: 0.04g

METHOD B - Thin-Layer Chromatography

- o TITANIUM CHROMOGENIC REAGENT FOR PHENOLIC COMPOUNDS ON THIN-LAYER PLATES
- o J. Chromatogr. 1978, 150(1), 293-294
  N. A. M. Eskin and C. Frenkel

- o Phenolic compounds are detected on thin-layer plates (silica gel G) by spraying with the chromogenic reagent, 20% titanium tetrachloride in concentrated hydrochloric acid. For resorcinol, an orange-yellow color develops immediately.
- o Limits: 4 µg

METHOD C - Wet Chemical and Gas Chromatography

- o THE DETERMINATION OF PHENOLS IN AQUEOUS EFFLUENTS
- o Water Res. 1973, 7(9), 1375-1384
  R. L. Cooper and K. C. Wheatstone

- o Phenol, cresols, xylenols, ethylphenols, and dihydric phenols are separated by gas-liquid chromatography after conversion to their trimethylsilyl ether derivatives. A flame ionization detector is used with a column of 5% tri-2,4-xylenyl phosphate on Chromosorb W. (AW-DCMS).
- o Limits: 0.1 mg/l.

Cross References - See cresol, method A; dinitrobenzene, A; phosphoric acid, A.

STRYCHNINE

METHOD A - Gas Chromatography

- A NEW, RAPID GAS CHROMATOGRAPHY METHOD FOR THE DETECTION OF BASIC DRUGS IN POSTMORTEM BLOOD, USING A NITROGEN PHOSPHORUS DETECTOR. PART 1. QUALITATIVE ANALYSIS
- J. Anal. Toxicol. 1978, 2 (1), 26-31
  W. O. Pierce, T. C. Lamoreaux, and F. M. Urry

- Basic drugs are determined in blood by extraction into n - butyl chloride and analysis by gas chromatography with a nitrogen phosphorus detector. Retention data are given for two columns, 3% OV-1 and 3% OV-17, on 100-120 mesh Chromosorb W.
- Limits: 200-500 ng; 1 ml blood sample

METHOD B - Liquid Chromatography

- HIGH-SPEED LIQUID CHROMATOGRAPHY OF ALKALOIDS. I.
- J. Chromatogr. 1974, 100 (1), 227-230
  R. Verpoorte and A. B. Svendsen

- Strychnine is subjected to high-speed liquid chromatography on Merckosorb Si 60, with detection at 254 nm. Retention times are reported for six solvent systems.
- Limits: 25 ng

METHOD C - Wet Chemical and Atomic Absorption Spectrometry

- THE INDIRECT ATOMIC ABSORPTION SPECTROMETRIC DETERMINATION OF SEVERAL ORGANIC BASES USING MOLYBDOPHOSPHORIC ACID
- Microchem. J., 1975, 20 (4) 468-475
  S. J. Simon and D. F. Boltz

- Complexation of strychnine or quinoline with molybdophosphoric acid and subsequent liberation of molybdate permits indirect determination of the bases by measuring absorbance of molybdenum at 313 nm in an air/acetylene flame.
- Limits: quinoline, 2 ppm; strychnine, 0.65 ppm

STYRENE

- vinylbenzene, phenylethylene, styrol, styrolene, cinnamene, cinnamol

METHOD A - Gas Chromatography

- GAS CHROMATOGRAPHIC DETERMINATION OF SELECTED ORGANIC COMPOUNDS ADDED TO WASTEWATER
- Environ. Sci. Technol. 1975, 9 (6), 588-590
  B. M. Austern, R. A. Dobbs, and J. M. Cohen
- Eleven organic compounds are recovered from wastewater by extraction with Freon, concentrated, and analyzed by gas chromatography (hydrogen flame ionization detector) on a column of 15% Carbowax 4000 on 80-100 mesh Chromosorb W(HP).
- Limits: 0.5 ng styrene

METHOD B - Wet Chemical and Gas Chromatography

- GAS CHROMATOGRAPHIC DETERMINATION OF STYRENE AS ITS DIBROMIDE
- J. Chromatogr., Vol. 136, No. 1, pp. 95-103 (1977)
  Y. Hoshika
- Styrene from air is collected in a cold trap (liquid oxygen), extracted with hexane, and brominated with bromine in chloroform. The dibromide is analyzed by gas chromatography with electron capture detection on a column of 3% OV-17 on 60-80 mesh Chromosorb W. Interference studies with 138 compounds are reported.
- Limits: 0.1-0.4 ppb from air

Cross References - See xylene, method C

2,4,5-T ACID

- 2,4,5-trichlorophenoxyacetic acid

METHOD A - Wet Chemical and Gas Chromatography

- SIMULTANEOUS DETERMINATION OF 2,4-DICHLOROPHENOXYACETIC ACID, 2,4,5-TRICHLOROPHENOXYACETIC ACID, AND 2-METHOXY-3,6-DICHLOROBENZOIC ACID IN SOIL AND WATER BY GAS CHROMATOGRAPHY WITH ELECTRON CAPTURE DETECTOR
- J. Agr. Food Chem. 1974, 22 (3), 453-458
  R. Purkayastha

- Soil and water samples of the above herbicides are extracted with diethyl ether, butylated with diazobutane, cleaned on a Florisil column, and analyzed by gas chromatography with electron capture detector. Columns providing good sensitivity and resolution are 11% (OV-17/QF-1) or 3% Carbowax 20M on Gas-Chrom Q or Chromosorb W, 80-100 mesh.
- Limits: 0.03-0.05 ppm in soil; no cleanup required for water samples

Cross References - See 2,4-D acid, method A; dicamba, A and C; dichlorvos, B; 2,4,5-T ester, A.

2,4,5-T ESTER

- o 2,4,5-trichlorophenoxyacetic esters

METHOD A - Resin Sorption, Wet Chemical, and Gas Chromatography

- o SIMPLIFIED RESIN SORPTION FOR MEASURING SELECTED CONTAMINANTS
- o J. Am. Water Works Assoc. 1976, 68 (4), 218-222
  G. A. Junk, J. J. Richard, H. J. Svec, and J. S. Fritz
- o 2,4-D, 2,4,5-T, and their esters are removed from water samples by XAD-2 resin and eluted with diethyl ether. One portion of the eluate is hydrolyzed and another is esterified with diazomethane. Toal chlorophenoxy herbicide is then determined by gas chromatography.
- o Limits: < 250 ng/l

Cross References - See chlorpyrifos, method A; 2,4-D ester, A; dichlone, A; dichlorvos, B; 2,4,5-T acid, A.

TETRAETHYL LEAD

- lead tetraethyl, TEL

METHOD A - Thin-Layer Chromatography and Visible Spectroscopy

- DETECTION AND DETERMINATION OF ALKYL LEAD COMPOUNDS IN NATURAL WATERS
- Water Pollut. Control (Maidstone, Engl.) 1977, 76(1), 123-128
  H. R. Potter, A. W. P. Jarvie and R. N. Markall

- Tetraethyllead is determined by extraction into petroleum ether, conversion to Et_2PbCl_2 (via iodine monochloride), extraction of the salt into water, and thin-layer chromatography against an authentic sample. The aqueous extract can also be converted to a colored product by reaction with pyridylazoresorcinol; the absorption of the resulting solution is measured at 515 nm.
- Limits: 2 mg/l of Et_2Pb^{2+} in the petroleum ether extract

METHOD B - Gas Chromatography and Atomic Absorption Spectrometry

- GAS CHROMATOGRAPHY - ATOMIC ABSORPTION SPECTROMETRY FOR THE DETERMINATION OF TETRAALKYLLEAD COMPOUNDS
- Anal. Chim. Acta 1976, 85(2), 421-424
  Y. K. Chau, P. T. S. Wong, and P. D. Goulden

- A combination of gas chromatography and atomic absorption spectrometry is used to determine tetraalkyllead compounds in which the alkyl substituents are methyl or ethyl. A column of 3% OV-1 on Chromosorb W is used to resolve the five organometallic compounds, and lead is detected at 217 nm. The response is linear to 200 ng of lead.
- Limits: 0.1 ng lead can be determined with certainty.

METHOD C - Gas Chromatography

- TEN NIOSH ANALYTICAL METHODS. SET 3.
- NTIS PB-275 834, 1977, 75-118.
  Stanford Research Institute

- A known volume of air is drawn through an XAD-2 tube to trap the organic vapors present. After desorption with pentane, tetraethyllead is determined by gas chromatography, with photoionization detection, on a column of 5% Carbowax 20M on 80/100 mesh Chromosorb W, AW. Dodecane or other suitable internal standard is used.
- Limits: 0.045-0.20 mg/m^3 (as lead)

TRICHLORFON

- Dipterex, Dylox

METHOD A - Wet Chemical and Gas Chromatography

- ROUTINE METHODS FOR ANALYSIS OF ORGANOPHOSPHORUS AND CARBAMATE INSECTICIDES IN SOIL AND RYEGRASS
- Pestic. Sci. 1977, 8 (4) 354-358
  P. T. Holland

- Trichlorfon and dichlorvos are extracted from soil, cooled to precipitate water and waxes, and analyzed by gas chromatography (alkali flame ionization detector) on a column of 3% SE-30 on 80/100 Gaschrom Q. On-column acetylation with acetic anhydride is employed for analysis of trichlorfon. Gas chromatographic results are confirmed by mass spectrometry.
- Limits: 0.2 ng

METHOD B - Plasma Chromatography

- PLASMA CHROMATOGRAPHY OF PESTICIDES
- J. Chromatogr. Sci. 1975, 13 (6), 285-290
  H. A. Moye

- Twenty-three pesticides and/or metabolites are analyzed at residue levels by plasma chromatography. Ion mobility spectra are presented and applications to analysis of liquid chromatographic fractions are discussed.
- Limits: 0.1 ng for trichlorfon, methyl parathion, 2,4-D, and carbaryl; 0.01 ng for parathion and ethion; 0.001 ng for diazinon and chlorpyrifos.

METHOD C - Wet Chemical and Gas Chromatography

- THE DETERMINATION OF TRICHLORFON IN WATER
- Fish. Mar. Serv. Res. Dev. Tech. Rep. 714, 1977, 1-14
  V. Zitko and D. B. Sergeant

- Trichlorfon is determined indirectly by hydrolysis to dichlorvos and analysis of the latter by gas chromatography and mass spectrometry. The pH of a solution of trichlorfon in acetone is adjusted by addition of sodium hydroxide solution. After 1.75 hr the hydrolysis is quenched by addition of sulfuric acid, and the mixture is extracted with ethyl acetate and concentrated. Gas chromatographic columns used are: 3% OV-1 on Chromosorb W, HP, 80/100 mesh with flame photometric detector; 4% SE-30 on Chromosorb W, HP, 100/200 mesh with electron capture detector.
- Limits: 1 µg/ℓ of trichlorfon in an aqueous sample.

Cross References - See chlorpyrifos, method A; coumaphos, A.

TRIETHYLAMINE

METHOD A - Wet Chemical and Thin-Layer Chromatography

- o A NEW SOLVENT SYSTEM FOR THE THIN-LAYER CHROMATOGRAPHIC SEPARATION OF THE DANSYL DERIVATIVES OF SOME BIOGENIC AMINES
- o J. Chromatogr. 1974, 90(1), 178-180
  G. C. Boffey and G. M. Martin

- o Twenty-two biogenic amines are derivatized with 1-naphthalene-sulfonyl chloride (dansyl chloride) and subjected to thin-layer chromatography on silica gel. The chromatograms are developed in the carbon tetrachloride - ethylene glycol monomethyl ether (85:15, v/v) solvent system, and fluorescence of the dansyl derivatives is observed at 366 nm.
- o Limits: 0.01-1.00 μM sample

Cross References - See butylamine, methods A and B.

VINYL ACETATE

- acetic acid ethylene ether

METHOD A - Gas Chromatography

- RHODIUM(II) CARBOXYLATES AS NEW SELECTIVE STATIONARY PHASES IN GAS-LIQUID CHROMATOGRAPHY
- Chromatographia 1972, 5(12), 301-304
  V. Schurig, J. L. Bear, and A. Zlatkis

- Dimeric rhodium(II) benzoate in squalane is employed as a stationary phase for gas-liquid chromatography of compounds containing oxygen functional groups. The carboxylate is coated on Chromosorb P, AW, DMCS, 100/120 mesh, and a flame ionization detector is used.
- Limits: not given

METHOD B - Wet Chemical

- MICRO DETERMINATION OF OLEFINIC UNSATURATION. REACTION OF N-BROMOSUCCINIMIDE WITH OLEFINS IN POLAR MEDIUM
- Fresenius' Z. Anal. Chem. 1972, 260(5), 359-361
  V. K. S. Shukla, U. C. Pande, and J. P. Sharma

- Olefinic unsaturation is determined by reacting a solution of the olefin in glacial acetic acid with a known excess of N-bromosuccinimide, which is back-titrated iodometrically. The reaction is rapid (10 minutes) and observes 2:1 stoichiometry in the polar medium.
- Sample size: 2-10 mg

Cross References - See acetaldehyde, method B.

XYLENE

- dimethylbenzene, xylol

METHOD A - Gas Chromatography

- SUSPENSIONS OF CRUDE OILS IN SEA WATER: RAPID METHODS OF CHARACTERIZING LIGHT HYDROCARBON SOLUTES
- Natl. Bur. Stand. (U.S.), Spec. Publ. 409, pp. 127-130 (1974)
  R. M. Bean

- Aqueous samples containing the xylenes are filtered to remove insoluble hydrocarbons, extracted with carbon tetrachloride, and analyzed by gas chromatography on a column of 4% Carbowax 20M TPA on 100-120 mesh Chromosorb W. o-Xylene appears as a separate peak, but m- and p-xylene are not resolved.
- Limits: 0.01 mg/l; sample size, 0.5 l

METHOD B - Gas Chromatography

- SOLVENTS IN SEWAGE AND INDUSTRIAL WASTE WATERS: IDENTIFICATION AND DETERMINATION
- Water Pollut. Control 1974, 73 (6), 656-672
  W. K. Ellison and T. E. Wallbank

- Aqueous samples of m-xylene undergo preliminary separation by steam distillation and extraction, followed by gas chromatography (electron capture detection; 10% Apiezon L on Chromosorb G, 60/80 mesh) and infrared and ultraviolet spectroscopy.
- Limits: recovery of 88% with 0.5 mg aliquot in 1-ℓ sample

METHOD C - Gas Chromatography

- GAS CHROMATOGRAPHIC SEPARATION OF STYRENE IN THE PRESENCE OF XYLENES AND PROPYLBENZENES
- Anal. Chem. 1974, 46 (14) 2225-2226
  D. M. Ottenstein, D. A. Bartley, and W. R. Supina

- For a mixture of nine compounds, including styrene and the three isomeric xylenes, base line resolution is obtained by gas chromatography on a column of 5% SP-1200 and 1.75% Bentone 34 on 100-120 mesh Supelcoport. Dual flame ionization detectors are used.
- Limits: a 0.10 µℓ injection of the mixture is well resolved

Cross References - See benzonitrile, method A; butyl acetate, C; dinitrobenzene, B; styrene, A.

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